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Securing Containerized Hazardous Wastes with Polyethylene Resin and Fiberglass Encapsulates

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SECURING CONTAINERIZED HAZARDOUS WASTES WITH POLYETHYLENE RESIN AND FIBERGLASS ENCAPSULATES

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This study investigates the fabrication and use of polyethylene resin and fiberglass to encapsulate and secure containerized hazardous wastes. Laboratory-scale encapsulates of composite structure were made from powdered, high-density polyethylene (HDPE) and epoxy-resin-wetted fiberglass. Methods are described for fabricating encapsulates with seamless polyethylene walls backed by fiberglass, and a demonstration is given of their high performance under severe leaching and mechanical stresses.

The objective of the study was to develop a method for securing hazardous wastes held either in small, corroding containers or in large ones such as 55-gal drums. The drums would be encapsulated on an as-is basis (i.e., their contents would not be emptied). Encapsulation would make them safe to transport and to deposit within a final disposal site such as a landfill.

This study is a companion to two other documents investigating the use of plastics for the encapsulation of corroding containers of hazardous wastes: Securing Containerized Hazardous Wastes with Welded Polyethylene Encapsulates (EPA-600/) and Securing Containerized Hazardous Wastes by Encapsulation with Spray-on/Brush-on Resins (EPA-600/).

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report presents results of investigations to develop and evaluate a unique method for making corroding, leaking containers of hazardous waste safe for disposal. Polyethylene resins and fiberglass materials were used to fabricate seam-free nonleaking encapsulates for the containers.

Francis T. Mayo, Director
Municipal Environmental Research
Laboratory

ABSTRACT

This study investigates the fabrication and use of polyethylene resin and fiberglass to encapsulate and secure containerized hazardous wastes. Laboratory-scale encapsulates of composite structure were made from powdered, high-density polyethylene (HDPE) and epoxy-resin-wetted fiberglass. Methods are described for fabricating encapsulates with seamless polyethylene walls backed by fiberglass, and a demonstration is given of their high performance under severe leaching and mechanical stresses.

The objective of the study was to develop a method for securing hazardous wastes held either in small, corroding containers or in large ones such as 55-gal drums. The drums would be encapsulated on an as-is basis (i.e., their contents would not be emptied). Encapsulation would make them safe to transport and to deposit within a final disposal site such as a landfill.

Cylindrical encapsulates 3 in. in diameter and 4 in. high were fabricated by employing heated, matched die molds. Fusion of powdered HDPE onto fiberglass casings yielded watertight encapsulates. They were characterized by seamless, 1/4-in.-thick HDPE walls backed by 1/50-in.-thick, fiberglass-reinforced epoxy resin. The fiberglass provided stiff surfaces of regular dimension that facilitated fabrication of HDPE walls and, in full-scale encapsulates, would also reinforce them suitably for manipulation, stacking, transportation, and final charging into a landfill.

Evaluations indicated that fiberglass-backed HDPE encapsulates assured high-performance retention of contaminants. Encapsulates exhibited stability in chemically aggressive waters, and they showed unusual resistance to rupture when compressed appreciably. The encapsulates examined held water soluble compounds of the following toxic metals: Ni, Cd, Hg, Cr, Zn, Cu, Sb, As, Se and Pb. In aqueous hydrochloric acid (an excellent solvent for metals), the hazardous consignments remained secure. When severely distorted vertically and laterally, the encapsulates not only withstood rupture, but unconstrained,they exhibited appreciable recovery of their original dimensions.

The high performance of laboratory-scale encapsulates indicated that full-scale encapsulates holding corroding 55-gal drums with hazardous wastes would keep their consignments secure when manipulated, transported, and sequestered within a landfill. Full-scale encapsulates with 1/4-in. polyethylene jackets backed by fiberglass fabricated according to this work are expected to satisfy Department of Transportation requirements for containers used to transport hazardous wastes.

This study is a companion to two other documents investigating the use of plastics for the encapsulation of corroding containers of hazardous wastes: Securing Containerized Hazardous Wastes with Welded Polyethylene Encapsulates (EPA-600/) and Securing Containerized Hazardous Wastes by Encapsulation with Spray-on/Brush-on Resins (EPA-600/).

This report was submitted in fulfillment of Contract No. 68-03-2483 by TRW Defense and Space Systems Group and Contract No. 68-03-2993 by Environmental Protection Polymers, Inc., under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period 1978 to 1980, and work was completed as of December 1980.

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ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

gal	--gallon
ml	--milliliter
µl	--microliter
L	--liter
in.	--inch
ft	--foot
mm	--millimeter
$in.^2$	--square inches
mm^2	--square millimeters
cc	--cubic centimeters
ft^3	--cubic feet
A	--angstrom
nm	--nanometer
g	--gram
lb	--pound
wt	--weight
pbw	--parts by weight
MPa	--megapascal
psi	--pounds per square inch
J/m	--joules per meter
BTU	--British thermal units
^oC	--degrees Celsius
^oF	--degrees Fahrenheit
N	--normality of solution
ppm	--parts per million
pH	$--\log [H_3^+O]$
min	--minute
h	--hour
sec	--second
ESCR	--environmental stress crack resistance
FRP	--fiberglass-reinforced plastic
AAs	--atomic absorption spectroscopy
UV	--ultraviolet
ht	--height
dia	--diameter
DOT	--Department of Transportation
PE	--polyethylene
HDPE	--high-density polyethylene

CHEMICAL SYMBOLS

Ni	--nickel
Cd	--cadmium
Hg	--mercury
Cr	--chromium
Zn	--zinc
Cu	--copper
Sb	--antimony
As	--arsenic
Se	--selenium
Pb	--lead
Sn	--tin
Cl_2	--chlorine
HCl	--hydrochloric acid
HNO_2	--nitrous acid
HNO_3	--nitric acid
H_2	--hydrogen gas
Ar	--argon
C_2H_2	--ethylene

SYMBOLS

--pump

--thermocouple

--valve

--metering valve

METRIC CONVERSION FACTORS

English system	Metric system
Gallon (U.S., 4 quarts)	3.785 liters
Inch	2.54 cm
Foot	30.48 cm
Yard	0.914 m
Square inch	6.452 cm^2
Square foot	0.092 m^2
Square yard	0.836 m^2
Cubic inch	16.387 cm^3
Cubic foot ($1,728\ in.^3$)	0.028 m^3
Cubic yard ($27\ ft^3$)	0.7646 m^3
^oF	-17^oC
Pound	
avdp. (16 dr)	453.592 g
troy (12 oz)	373.24 g
Ton	
long (2,240 lb)	1.016 t
short (2,000 lb)	0.907 t
Mile (1,760 yd)	1.609 km

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The authors acknowledge the contribution of Dr. David Pinkerton, who set forth a conceptual design of an apparatus for making large containers.

Our thanks are also extended to Mrs. V. R. Butler for providing clerical services.

SECTION 1

INTRODUCTION

This study investigates the fabrication and use of polyethylene resin and fiberglass to encapsulate and secure containerized hazardous wastes. The work is particularly concerned with securing corroding 55-gal* steel drums holding hazardous wastes. The term "securing" means to make containers safe for transporting to and sequestering within a direct disposal site such as a landfill. Three technical approaches are possible:

1) Placing containers as-is in receptacles and sealing the ensemble.

2) Applying material onto the walls of containers, thereby reinforcing them.

3) Removing the container contents and then managing them by predetermined procedures.

This work involves the first approach.** Work that took the second approach was carried out at TRW for EPA under Contract No. 68-03-2483. The status of this work was described earlier in the report entitled Study of Encapsulate Formation with Resins by Spray and/or Brush Under Atmospheric Conditions for Use in Management of Containerized Hazardous Wastes. †The third approach has been employed, for example, by the Mitre Corp. (under contract to the State of Massachusetts) for managing 55-gal drums with hazardous contents.

*The policy of the U.S. Environmental Protection Agency is to express all measurements in metric units. When implementing this practice will result in undue cost or lack of clarity, conversion factors are provided for the nonmetric units used in the report. Generally, this report uses English units of measure. For conversion to the metric system, see the conversion factors listed on p.xii.

**Simple scooping of the wastes into vehicles and transporting to and depositing within a landfill was discounted because of potential hazards in waste collection, requirement of special vehicles for transportation in compliance with safeguards established by the Department of Transportation for transporting hazardous wastes, and potential deleterious impact on the environment by unsecured hazardous wastes residing in a landfill.

† Twenty-Sixth Monthly Report, January 1979, Contract No. 68-03-2483.

Use of the second approach allowed employment of simple spraying techniques and commercially available apparatuses. But those advantages were offset by the difficulty of fabricating encapsulates with high performance mechanical properties. The third approach, in contrast to Approaches 1 and 2, yielded a desirable reduction of wastes for ultimate disposal, but it required opening the containers of hazardous contents. Such an operation exacerbates risk to personnel and the environment and makes necessary costly assays of waste to determine appropriate management options.

The first approach--the sealing of corroding containers of hazardous contents in receptacles--is an instinctive and elementary approach to waste management that has been used previously in commercial operations. Unfortunately, the wastes were not adequately secured, because the receptacles employed did not exhibit sufficient performance for satisfying Department of Transportation regulations and/or safeguarding hazardous consignments under landfill environmental conditions. These receptacles were steel containers of about 85-gal capacity constructed similarly to steel 55-gal drums and, as such, subject to corrosion. (They are presently employed as receptacles for leaking waste from corroding 55-gal drums.)

The size and construction of commercial plastic receptacles that exhibit corrosion resistance (such as polyethylene 55-gal drums) were not suitable for securing 55-gal metal drums. The largest commercial plastic vessels that may be transported with compliance to DOT regulations were 55-gal drums fitted with bung holes. Available wide-mouth drums did not qualify. Larger plastic vessels were not fitted with the means to effect secure closure. They were employed mainly as liners of steel and fiberglass-reinforced plastic vessels, or as free-standing receivers and holding tanks. Their value was particularly noteworthy in process and storage operations involving corrosive chemicals. There was great interest by plastics fabricators to make very large, free-standing chemical tanks from high-density polyethylene (HDPE) to replace plastic-coated or glass-lined steel tanks, stainless steel tanks, and fiberglass-reinforced plastic tanks.

Because of the absence of suitable commercial receptacles, this work established technical expertise for making the first approach a viable option for management of corroding 55-gal drums of hazardous consignments. A new type of receiver for drums was set forth that was of composite construction characterized by seamless, HDPE outer walls reinforced by fiberglass casing. The container encased wastes within a chemically stable encapsulate having high mechanical performance, and it was geared to overcome deficiences in state-of-the-art plastic containers created largely by the use of lids, gaskets, sealants, threading, and hoops for the making of closures.

The technique for making such encapsulates was based on the results of previous work performed by TRW for EPA under Contract No. 68-03-2037. The previous work is described in EPA 600/2-77-045 (August 1977), "Development of Polymeric Cementing and Encapsulating Process for Managing Hazardous Wastes." This project concerned management of unconfined waste

particulates by fashioning them into agglomerates and fitting them with seamless jackets of 1/4-in.-thick HDPE. The resulting agglomerates stiffened the flexible plastic outer walls of the encapsulates, and thereby the encapsulates maintained their dimensional integrity under appreciable compression. In the present project, the stiffening role is carried out by fiberglass casings.

This report describes the technique for encapsulate fabrication on a laboratory scale and outlines the product performance. The technique employed here may appear as essentially the same as that developed previously for managing unconfined, particulated wastes, but that is not the case. The technique described here was found to be preferable after investigations of a number of other options, which are discussed in the Appendices. The Appendices also contain additional information about the preferred encapsulation fabrication technique.

SECTION 2

CONCLUSIONS

Watertight, laboratory-scale encapsulates of hazardous wastes can be readily fabricated from powdered polyethylene and fiberglass. Seamless polyethylene plastic walls backed by fiberglass casings were the structural features of the encapsulates. Their high performance under applied chemical and mechanical stresses was due to the material homogeneity of their outer walls and to their composite structure. (The encapsulates viewed as containers, in our opinion, were unique, and their performance was estimated to be significantly in advance of that of state-of-the-art containers fabricated from low-in-cost materials. Such encapsulates scaled to encase corroding 55-gal drums holding hazardous wastes were expected to comply to Department of Transportation criteria governing containers employed in transporting hazardous wastes and to hold their contents secure under landfill conditions.)

Specifically, the work gave rise to the following observations and suppositions:

- Encapsulates of laboratory-scale size consisting of 1/4-in.-thick high-density polyethylene, seamless jackets backed by 1/50-in. fiberglass casings showed high performance retention of enclosed w stes when subject to severe aqueous leaching and mechanical stresses.
- Encapsulate properties reflected the functions of the fiberglass casings and the tough, flexible jackets. The casings facilitated ready fabrication of encapsulates by providing suitable substrates to carry out polyethylene fusion, and they conserved the dimensional integrity of encapsulates under mechanical stresses. The flexible jackets sealed the casings and prevented contact of the hazardous consignments with environmental leaching waters. When encapsulates were distorted by mechanical stresses such as impact and compressive ones wastes were safeguarded by jackets even though flaws occurred in the casings.
- Jacket formation by fusion of powdered polyethylene in matched die molds under minimal mechanical pressures yielded consolidated polyethylene exhibiting its full compliment of expected properties. Consequently, the jackets exhibited flexibility and toughness. The high elongation to break of the jackets prevented rupture

of the encapsulates even when they were severely distorted.

- Method of heating used in this work for jacket formation for making laboratory-scale encapsulates would not be appropriate for making full-scale encapsulates. Convenience and simplicity in producing small specimens dictated this method as the one most suitable for the task at hand. But it required appreciable time for formation of jackets due to the slow heat exchange rate. A more time effective method would be realized by applying the heating elements directly upon the mold walls. With commercial rotomolding practice as a guideline, it should be possible to fuse the resin in several minutes.
- Appreciable amounts of unconfined volatile liquid present did not preclude sealing of the encapsulates. Thus liquids accumulating from a leaking 55-gal drum in a full-scale encapsulate under fabrication should not interrupt the operation. Sealing the hazardous consignments required fusing resin only at the extremity of the encapsulate. Thus no heating of the encapsulate consignment was anticipated under these conditions.
- Encapsulate fabrication by means of this work indicated that heavy duty equipment would not be required for making full-scale encapsulates. This feature was realized because powder rather than molten resin was employed in charging the molds. (In injection and extrusion molding the movement of molten resin makes necessary heavy-duty equipment for managing the required resin displacement pressures.) Therefore full-scale encapsulates could be fabricated by readily transportable equipment.
- The work indicated that corroding 55-gal drums holding hazardous wastes can be managed as follows:

    1) Transport equipment and materials (materials can also be in the form of partially fabricated encapsulates) to the site where drums reside.

    2) Secure drums in fiberglass-backed polyethylene encapsulates and transfer them to a landfill (encapsulates secure contents in case of mishaps in transportation).

    3) Charge encapsulates into a landfill according to accepted practice with respect to disposing containerized wastes.

SECTION 3

RECOMMENDATIONS

This work should be advanced to fabricating and characterizing full-sized encapsulates of about 65-to 85-gal capacity. (This size range was estimated to be that most applicable to managing viable 55-gal drums and those distorted or misshapen.) Fabrication would consist of:

1) Forming cup-like casings/employing commercial techniques of spray-up or hand lay-up of fiberglass.

2) Fusing powdered polyethylene upon the walls of the cup-like casing to obtain a receiver for insertion of a 55-gal drum.

3) Sealing the receiver with additional fiberglass and then fusing additional polyethylene onto the uncoated casing.

The product would be a double-walled encapsulate having a 1/4-in.-thick polyethylene seamless outer wall backed by approximately 1/8-in.-thick reinforcing fiberglass casing. The outer wall thickness will be the same as that utilized and evaluated in this work. The casing thickness is an estimated value; a more exact value would stem from analysis of stresses an encapsulate would encounter in manipulation, stacking, transportation, and sequestering in a landfill.

An apparatus should be constructed for making full-scale encapsulates. It would carry out in-place fusion and solidification of powdered polyethylene. The apparatus would not be encumbered by sophisticated mechanical equipment because the resin is charged into the apparatus in powdered form at atmospheric temperatures. The process is analogous to commercial rotomolding. (A feature of these molding processes is that it yields plastic products that are essentially stress free. However, rotomolding is not used extensively in commercial practice because it is viewed to be a slow process with respect to injecting, extruding and blowing, the latter employed commercially for making 55-gal plastic drums. But encapsulation of 55-gal steel drums in the field is viewed to be inconsistent with most commercial practices in plastic product fabricating. One aspect is the rate of product formation in the former case will be determined in great part by the time required for manipulating the containerized wastes. This time span is assumed to be appreciably greater than the time span of molding, whereas in the latter cases the rate of product formation depends in most cases upon its residence time in the mold.)

The apparatus for making full-scale encapsulates should be similar to that illustrated in this study for making laboratory-scale encapsulates, but with more facile heating arrangements. Nevertheless, that required for making full-sized encapsulates will be of a simple nature when compared to commercial plastic product fabricating devices. It would be comparable to devices employed commercially in rotomolding which are simple in comparison to other plastic product fabricating devices. Yet the apparatus needed for encapsulation would be even more simple because it would be stationary (i.e., no provision would be required for rotating and tumbling as in rotational devices).

Characterization of full-scale encapsulates in the initial phase should follow the procedure given in this work for laboratory-sized encapsulates. With demonstration of satisfactory resistance to leaching and high mechanical performance, further characterization of encapsulates should mainly concern noting their performance under real-life stresses. These would stem from encapsulate fabricating, manipulating, stacking, and locating within a final disposal site such as a landfill.

SECTION 4

MATERIALS

This section sets forth new types of containers for use in securing containerized hazardous wastes. They were structured to encapsulate hazardous wastes existing in containers which were undergoing deterioration. Of particular concern was the fate of hazardous wastes in corroding 55-gal drums. The containers consisted of homogeneous, flexible outer walls and rigid, inner walls. The outer walls secured the hazardous consignments to chemical and mechanical stresses. And the inner ones provided rigidity for stacking, transporting, and depositing containers in a final disposal site.

CANDIDATE OUTER WALL MATERIALS

The polyolefins were selected as the materials for the outer walls, particularly high-density polyethylene. On a price/performance basis, polyolefins were unique. Bitumens and asphaltics although of lower cost did not exhibit engineering properties required in containers. And engineering resins such as the thermoplastic polyesters were appreciably more costly than the polyolefins.

Polyethylenes in the market place were many types; the types preferred in this work were those employed in commercial rotomolding of polyethylene containers. In our opinion, all these types were satisfactorily employable in making the containers of this work. Other types employed in extrusion and blow molding were also applicable because successful in-place fusion of polyethylene particulates as carried out in this work was found to be relatively resin-viscosity insensitive. Low pressure, linear polyethylenes were being made available commercially, and these types claimed to exhibit advanced performance were also suitable candidates. Furthermore, particulated scrap polyethylene was considered in order to reduce costs; yet functional containers were anticipated because mechanical stresses encountered by containers of this work were expected to be appreciably less severe than those affecting large, commercial, polyethylene containers.

CANDIDATE INNER WALL MATERIALS

Fiberglass was selected for the inner walls because it is lightweight, rigid and low-in-cost. Concrete was considered due to its potential lower costs, but this advantage was seen to be mitigated by the additional weights required to obtain tough structures. Various types of fiberglass existed, and the lowest cost varieties such as chopped glass fiber employed in spray-

up of plastic shower stalls, tubs, etc., were preferable. Cloth, mat and roving were eliminated in that order due to their greater costs. Nevertheless, mat and roving were seen to be viable condidates in situations where processing advantages were being sought. In preparing laboratory-scale encapsulates, cloth was employed because it facilitated encapsulate fabrication.

LABORATORY-SCALE ENCAPSULATE TEST SPECIMENS

Fiberglass-reinforced polyethylene encapsulate test specimens were produced in the laboratory to demonstrate their high chemical and mechanical performance. They were cylindrically shaped, 3 in. in diameter and 4 in. high, and they had walls of two-layer construction. The walls consisted of about 1/4-in.-thick polyethylene jacket, backed by 1/50-in.-thick fiberglass casing.

Some specimens contained sand, and some were hollow. The sand-filled specimens were charged with aqueous solutions of heavy metals to simulate loss of container contents within encapsulates. They were then examined with respect to their ability to secure metal contaminants under harsh aqueous leaching conditions. (Sand and soil were contemplated for use to fill voids between containers and container/encapsulate walls. Compaction of containerized wastes rather than filler utilization was considered potentially desirable because it gives rise to more effective use of encapsulating materials and product manipulation methods. But compaction operation was also seen to require pollution control procedures that may be burdensome, hazardous and costly. Thus the containers were initially tailored to manage hazardous waste containers on an as-is basis.)

The hollow specimens, mainly, were employed to evaluate the mechanical performance of encapsulates. It was assumed that a clearer picture of encapsulate wall performance would be realized by more detailed examinations of encapsulates without filler than those with filler.

Nature of Commercial-Scale Encapsulates

A commercial-scale encapsulate was envisioned to provide about 75-gal capacity. The walls would consist of a 1/4-in.-thick polyolefin jacket backed by a 1/16- to 1/8-in.-thick chopped fiberglass casing. The jacket and the casing make up about 3.5% and 1.8% of the encapsulate volume, respectively. The exact thickness and nature of the fiberglass moiety in commercial-scale containers were to be determined in future work for encapsulates to withstand the mechanical stresses they may encounter in real-life manipulation and final disposition.

SECTION 5

ENCAPSULATE FABRICATION

Laboratory-scale test specimens were prepared for use in determining performance of double-wall containers. Since the outer walls were expected to ultimately secure the hazardous consignments, the test specimen wall thickness, 1/4-in., was of the same dimension as that contemplated for use in commercial-scale containers. The nature of the load-bearing inner walls would be determined in a later program in evaluations of full-scale test specimens.

Fabrication of a test specimen was begun by pouring construction sand in the glass/resin casing (Figure 1) and encasing the ensemble (Figure 2).* Fusion of polyethylene was then carried out in the annulus resulting from positioning an encased product in the mold (Figure 3). The encased products were then submerged in powdered polyethylene (Figure 4)** and the polyethylene was fused (Figure 5).

The steel mold walls were heated by hot mold platens with the rise in temperature of the mold walls monitored by a thermal probe. The changes in phase of the polyethylene (i.e., powdered resin to molten resin, and subsequently to solidified resin with cooling of the mold walls) was readily observed on the pressure gauge. The speed of jacket formation was essentially limited only by the heat exchange rate. (Commercially, 1/4-in.-thick polyethylene products were fabricated in 30 sec.)

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*For convenience, an epoxy formulation was employed to stiffen the glass. It consisted of DEN438 (Dow Epoxy Novalac), Methyl Nadic Anhydride, N,N-Dimethylbenzylamine, and acetone in parts by weight 56.9, 28.9, 1.7, and 12.5, respectively. The Methyl Nadic Anhydride was added to a solution of the remaining ingredients at time of use. The formulation-wetted glass was air dried to remove the acetone and then heated for 3 to 4 hours at 300°F. The resin content of the glass was about 20% by weight.

For commercial-scale containers, the lower-cost thermosetting polyester resins would be preferred to the epoxy ones.

**Marlex TR 955 from Phillips Petroleum.

Figure 1. Fabrication step of glass/resin encasement of sand.

Figure 2. Glass/resin-encased product.

Figure 3. An encased product centered in mold for resin encapsulation.

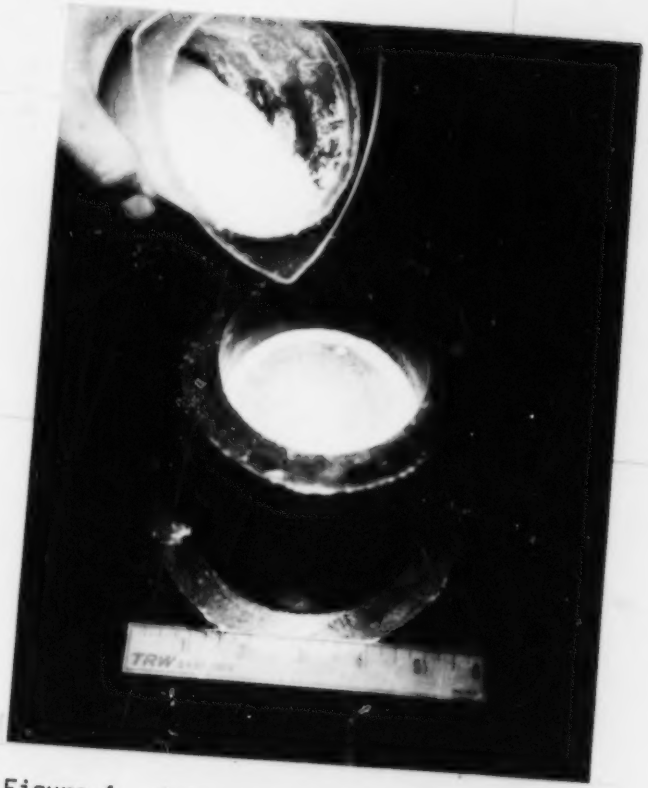

Figure 4. Submerging encased product with powdered polyethylene.

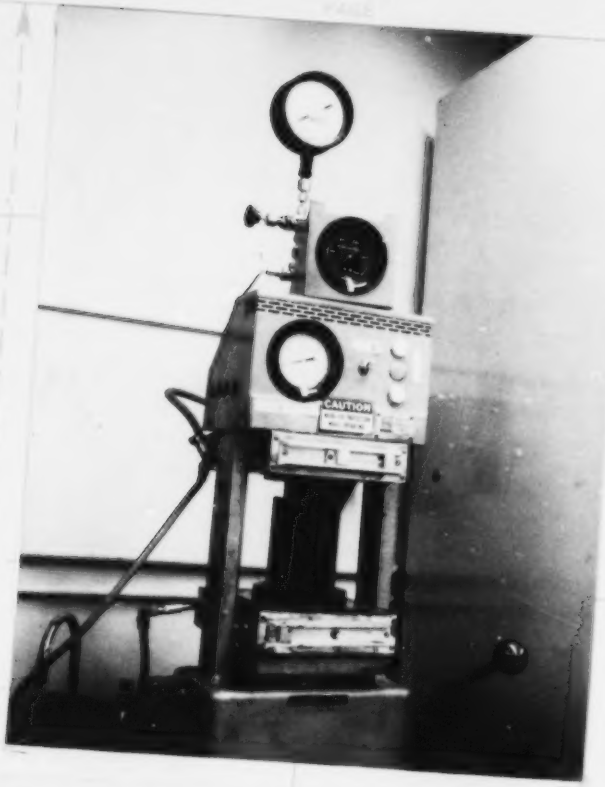

Figure 5. Apparatus employed for fusing polyethylene.

The specimen resulting from the fusing operation (Figure 5) is shown in Figure 6. Figure 7 shows the injection of the solution of hazardous chemical agents into the cavity of the product (the solution occupies the free space between the sand particles).

Encapsulation of the test specimen was completed with the use of additional polyethylene powder (Figure 8). The following metal contaminants were localized in the specimens: Ni, Cd, Hg, Cr, Zn, Cu, Sb, As, Se and Pb. The specimens were placed in plastic containers holding 1,500 cc of leaching solution. (Plastic, in contrast to glass, allows ready agitating and tumbling of the ensembles.) Figure 9 shows the type of container used to hold the specimen during the process. A specimen residing in the container is shown in Figure 10, and the set of test specimens produced is shown in Figure 11.

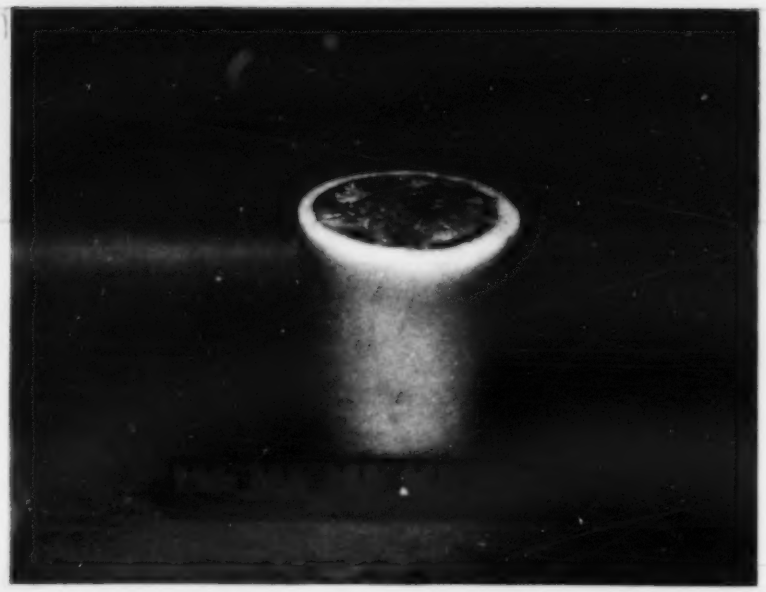

Figure 6. A polyethylene-encased product stemming from the first fusion step.

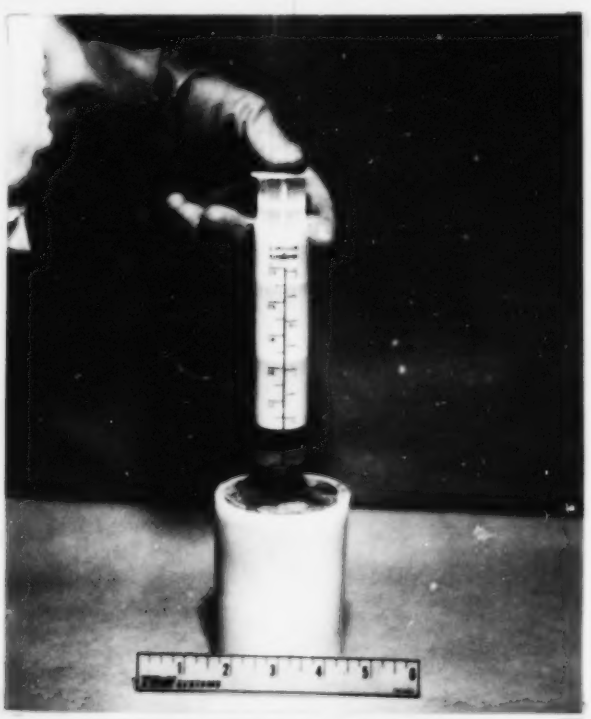

Figure 7. Hazardous chemical agents being injected into the test specimen.

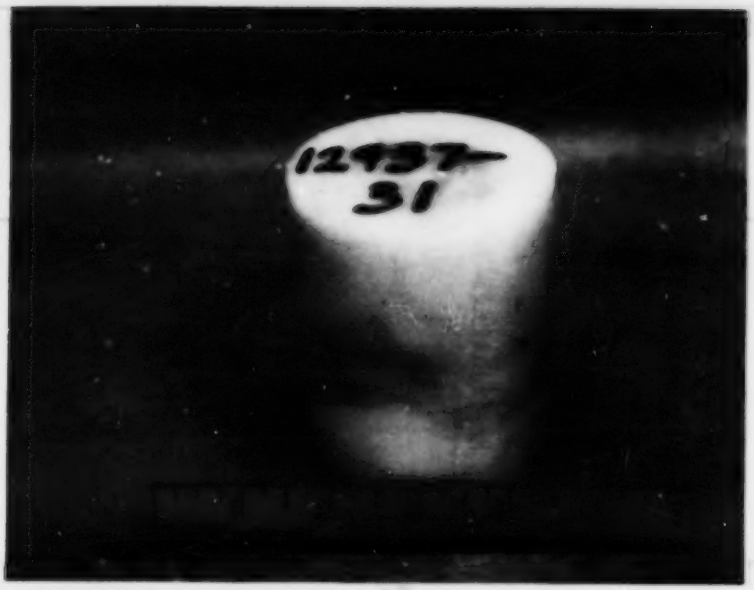

Figure 8. Completed polyethylene-encapsulated test specimen.

Figure 9. Container holding specimen in leaching solution.

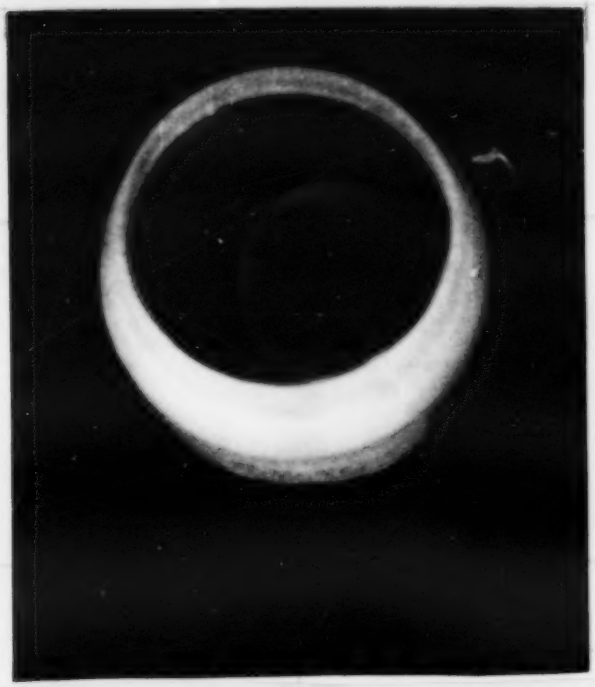

Figure 10. Specimen in container holding leaching solution.

Figure 11. Set of specimens prepared for determining leaching performance.

SECTION 6

ENCAPSULATE PERFORMANCE EVALUATIONS

Encapsulated containers molding contaminants were expected to remain functional under stresses they may encounter in manipulation, transportation and final disposition. This performance of test specimens in selected laboratory tests shown here was seen to be indicative of the performance capability of commercial-scale encapsulates. Particular emphasis was placed upon estimating the ability of specimens to withstand rupturing under high applied loads and to hold the contaminants secure when leached by water and acidic aqueous solutions.

It was very important to show first that the encapsulating process fashioned a tough, flexible phase, i.e., a polyethylene jacket that exhibits properties that were expected of the resin. (The nature of processing can affect final resin properties. Polyethylene products were fabricated commercially by different techniques: rotational molding, extrusion and injection molding. In this work the products were fashioned by heating polyethylene powder in place.) The evidence provided here shows that polyethylene jackets fashioned by powder fusion exhibited the mechanical properties that the condensed resin was expected to show.

The mechanical evaluations showed that fiberglass-casing-reinforced polyethylene containers will undergo appreciable distortion under very high mechanical compressive stresses without rupture and consequent loss of their contents. This phenomenon was due to the feature which characterized their compositions, i.e., seam free outer walls which were tough and flexible. The casings were rigid so that high mechanical loads may be withstood by containers without structural distortion; thus resulting large encapsulates with nonuniformly distributed internal contents should maintain their dimensional integrity under manipulation and final disposition conditions.

The permeability data indicated that polyethylene encapsulates would effectively prevent diffusion of hazardous materials under severe leaching conditions. Essentially no heavy metals were present in the leaching solutions into which encapsulates were submerged and in which they were occasionally tumbled. This determination was made from analysis for metals in the leaching solutions. The analytical determinations were carried out by employing the Jarrell-Ash Atomic Absorption Spectrophotometer, model 850.

SPECIMENS UNDER TENSIVE AND COMPRESSIVE STRESSES

The mechanical performance of encapsulates was characterized by observations and characterizations of specimens under tension and compression. The tensile determinations allowed comparison of values of polyethylene jackets fused upon fiberglass casings to those given in the literature for this resin. The compressive determinations examined the influence of contents upon the compressive nature of encapsulates. For this purpose, the following types of specimens were fabricated: hollow ones, and specimens filled with construction sand. Hollow specimens allowed determinations of inherent strengths of encapsulates and amplified the influence of encapsulate wall structure. Sand was one material recommended for filling space interstitially in encapsulates holding containers, thus it was desirable to observe its effect.

Tensile Specimens and Properties

The walls of encapsulates were sectioned and tensile specimens were fashioned from the sectioned walls. Figure 12 shows a tensile specimen; the darkened area was due to the reinforcing casing. The specimen was mounted in the jig of tensile apparatus (see Figure 13). The apparatus and extended specimen are shown in Figure 14.

The stress-strain graph showing the response mode of encapsulate walls upon extension is given in Figure 15. A distinctive feature of this graph was the high initial slope of the curve and the sharp break at about 2-1/2% elongation with precipitate loss in tenacity. This loss, however, was minimal (a brittle material would exhibit catastrophic failure). The tensile strength then increased to a maximum value at about 15% elongation and then descended to a residual value of approximately half that at the maximum and maintained that value until break point (about 650% elongation).

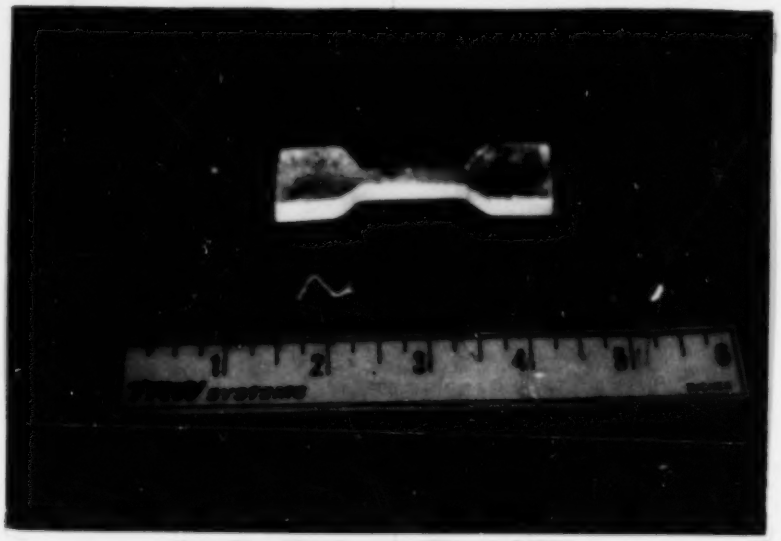

Figure 12. Tensile specimen.

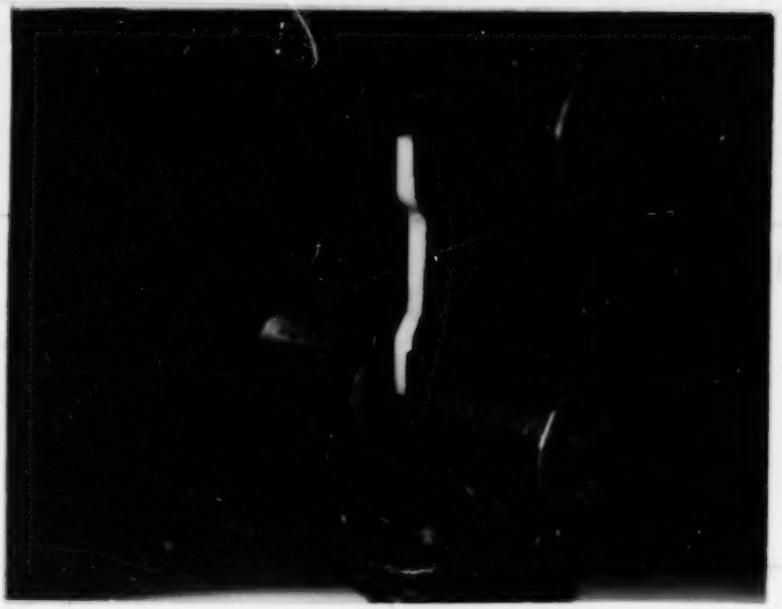

Figure 13. Tensile specimen in jig.

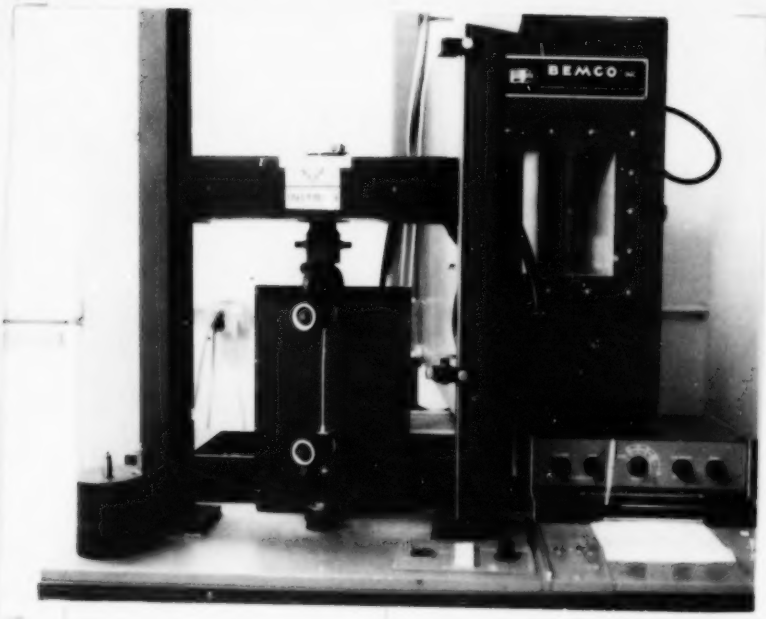

Figure 14. Tensile apparatus and typical extended specimen.

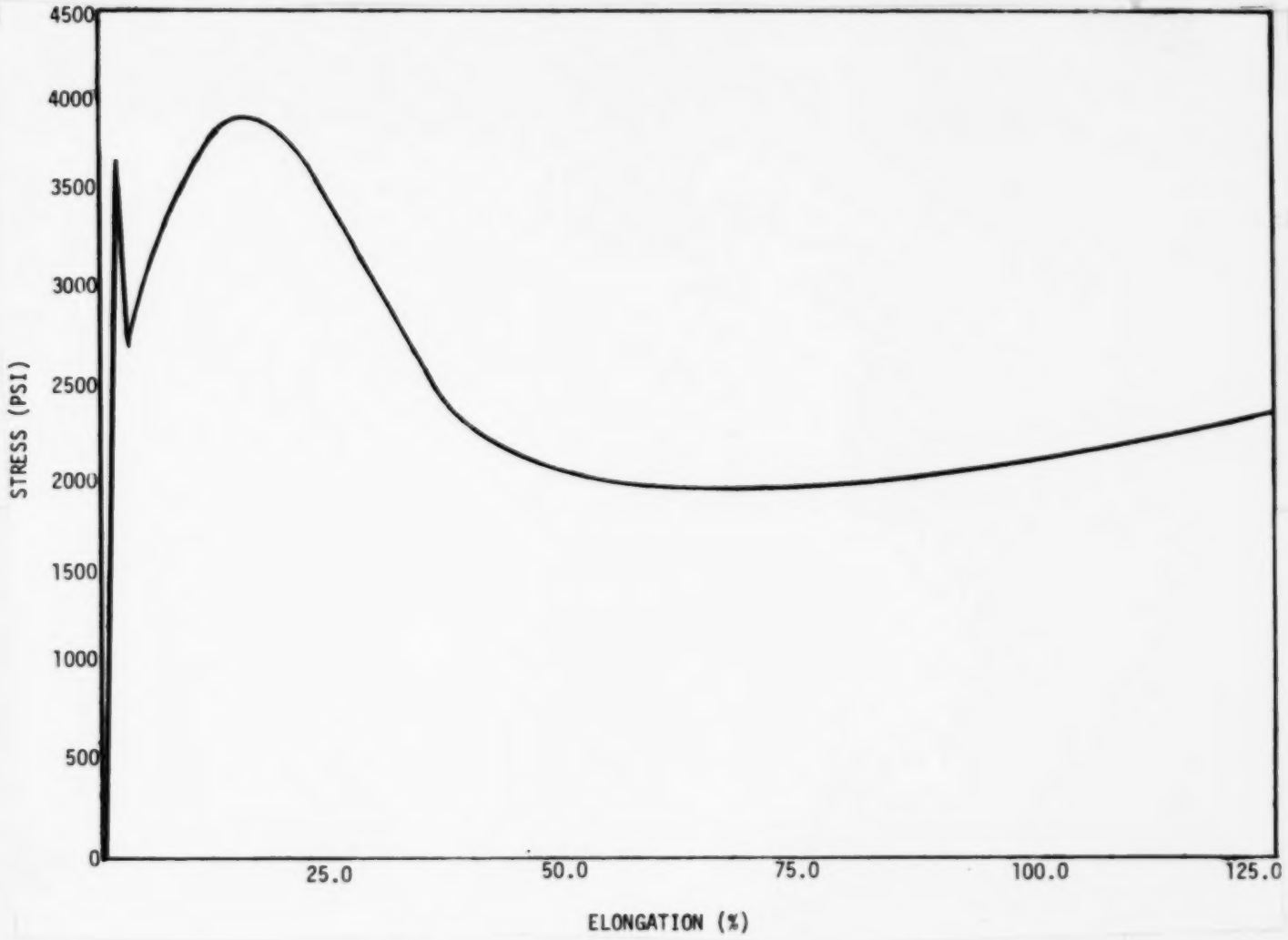

Figure 15. Stress-elongation curve for polyethylene with reinforcement.

Table 1 gives average values for tensile specimens characterizing their mechanical response. The data reflected the effect of the fiberglass casing and the polyethylene jacket upon tensile properties. The casing contributed rigidity in initial extension. With failure of the casing, the tensile properties reflected those of condensed polyethylene.

To compare the tensile properties of encapsulate walls to that of unsupported polyethylene, a 1/4-in. sheet of polyethylene was molded under the same condition employed for fashioning polyethylene jackets into encapsulate casings.

Figure 16 shows a tensile specimen stamped from the sheet positioned in the tensile apparatus jig.

Figure 17 shows the stress-strain graph for polyethylene that was not reinforced. One notices the absence of the spiked portion of the curve seen in Figure 15. Table 2 lists average values for tensile specimens which characterize their mechanical response.

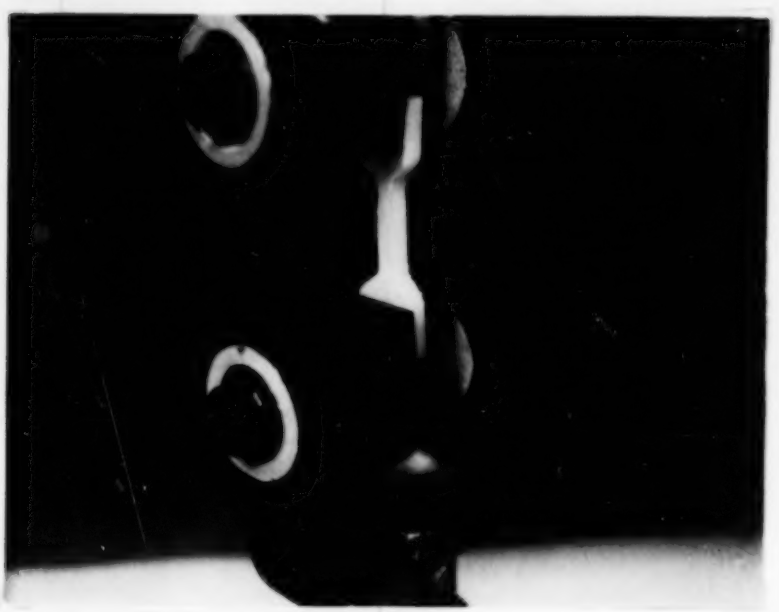

Figure 16. Polyethylene specimen positioned for testing in jig of tensile apparatus.

TABLE 1. TENSILE PROPERTIES FOR POLYETHYLENE SPECIMENS BONDED TO GLASS-RESIN REINFORCEMENT*

Properties	International System of Units (S.I.)		English System	
	Value	Unit	Value	Unit
Reinforcement Modulus	1380 ± 13.8	MPa	200000 ± 2000	psi
Tensile Strength @ 1st Yield (based on total cross-sectional area)	19.4 ± 5.1	MPa	2820 ± 740	psi
(based on cross-sectional area of glass)	126 ± 25	MPa	18300 ± 3600	psi
Extension @ First Yield	2.8 ± 1.5	%	2.8 ± 1.5	%
Stress Transfer	17.2 ± 1.4	MPa	2490 ± 200	psi
Extension @ Stress Transfer	4 ± 1	%	4 ± 1	%
Tensile Strength @ 2nd Yield	24.3 ± 4.2	MPa	3520 ± 610	psi
Extension @ 2nd Yield	18 ± 3	%	18 ± 3	%
Modulus @ 3% Extension	172 ± 13.8	MPa	25000 ± 2000	psi
Tensile Strength @ Break (based on original cross-sectional area)	20.2 ± 3.2	MPa	2923 ± 462	psi
Extension @ Break	610 ± 100	%	610 ± 100	%

*Values given as arithmetic means of five samples with their respective standard deviations, References 1 and 2.

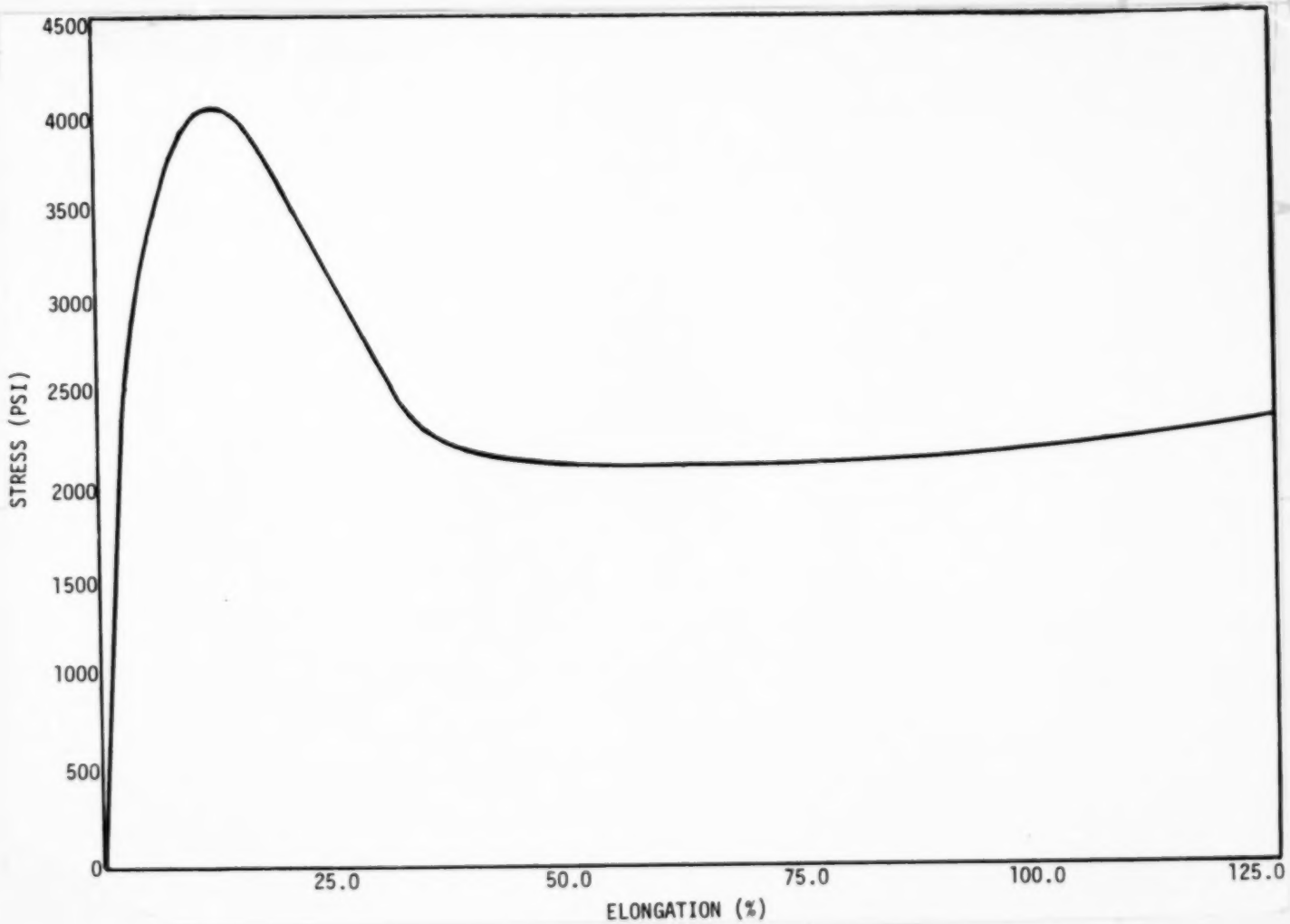

Figure 17. Stress-elongation curve for polyethylene without reinforcement.

TABLE 2. TENSILE PROPERTIES FOR POLYETHYLENE SPECIMENS WITHOUT REINFORCEMENT*

Property	International System of Units (S.I.) Value	Unit	English System Value	Unit
Elastic Modulus in Tension	930 ± 210	MPa	135,000 ± 30,000	psi
Modulus @ 3%	190 ± 21	MPa	27,000 ± 3,000	psi
Tensile Strength @ Yield	26.8 ± 2.4	MPa	3,890 ± 350	psi
Extension @ Yield	16 ± 2	%	16 ± 2	%
Tensile Strength @ Break (based on original cross-sectional area)	23.4 ± 2.1	MPa	3,390 ± 310	psi
Extension @ Break	680 ± 120	%	680 ± 120	%

*Values are arithmetic means of five samples with their respective standard deviations, References 1 and 2.

The initial portion of the curve of Figures 15 and 17 are amplified for comparative purposes in Figure 18. Notice the significant contribution of the casing to the initial modulus and tenacity even though it does not constitute a significant portion of the cross-sectional area of the casing-polyethylene composite. The casing-polyethylene curve extrapolated to the origin approximately tracks the curve resulting from unsupported polyethylene.

The tensile properties of polyethylene obtained in this work were compared to those provided in the literature for this material in Table 3. The table showed that the nature of the resin was the same whether fused upon casing, powder fused alone, or characterized by the vendor.

Additional data concerning polyethylene employed in this work are given in Table 4. The table was constructed from data given in two sources: the vendor, Phillips Petroleum, and Reference 3.

Compressive Specimens and Properties

The nature of cylindrical polyethylene encapsulates under compressive stresses were investigated with respect to mechanical loads applied to specimens in vertical and lateral positions. Figures 19, 20, 21, 22 and 23 show the response of the vertical encapsulate to progressively increasing compressive stress. The high degree of compression observed was characteristic of all specimens under vertically applied load. The nature of the mode of response of the specimens to the applied load differed: hollow encapsulates developed two bulges (2nd order buckling) as shown in Figure 24, and sand-filled encapsulates developed single bulges (1st order buckling) as shown in Figure 25. No explanation was on hand that accounted for these two observed modes of deformation.

Figures 26 and 27 show the response modes of encapsulates under lateral compression. Under extreme degree of compression no rupture of the encapsulates was observed. The nature of the encapsulates resulting from lateral compression are shown in Figures 28, 29 and 30. (The "circles" on the compressed encapsulates stemmed from the imprints on the faces of the compression rods.)

The ability of the encapsulates of this work to undergo extreme lateral compression without failure was a feature of these structures that was not, in our opinion, characteristic of conventional containers. (Conventional containers fitted with lids will not undergo appreciable lateral compression without rupture of the lid/container wall interface. With respect to maintaining a watertight configuration, even minimal degrees of compression would be detrimental.)

Figure 31 shows graphically the response of hollow encapsulates under compressive loads. The influence of the reinforcement due to the casing is clearly noted on the graph. The spiked initial portion of the curve, enlarged in Figure 32, was similar to that characterizing brittle materials such as fiberglass-reinforced epoxy composites in compression to failure. In contrast to such materials which undergo catastrophic failure, the encapsulates underwent minimal loss of load-bearing properties as a result of

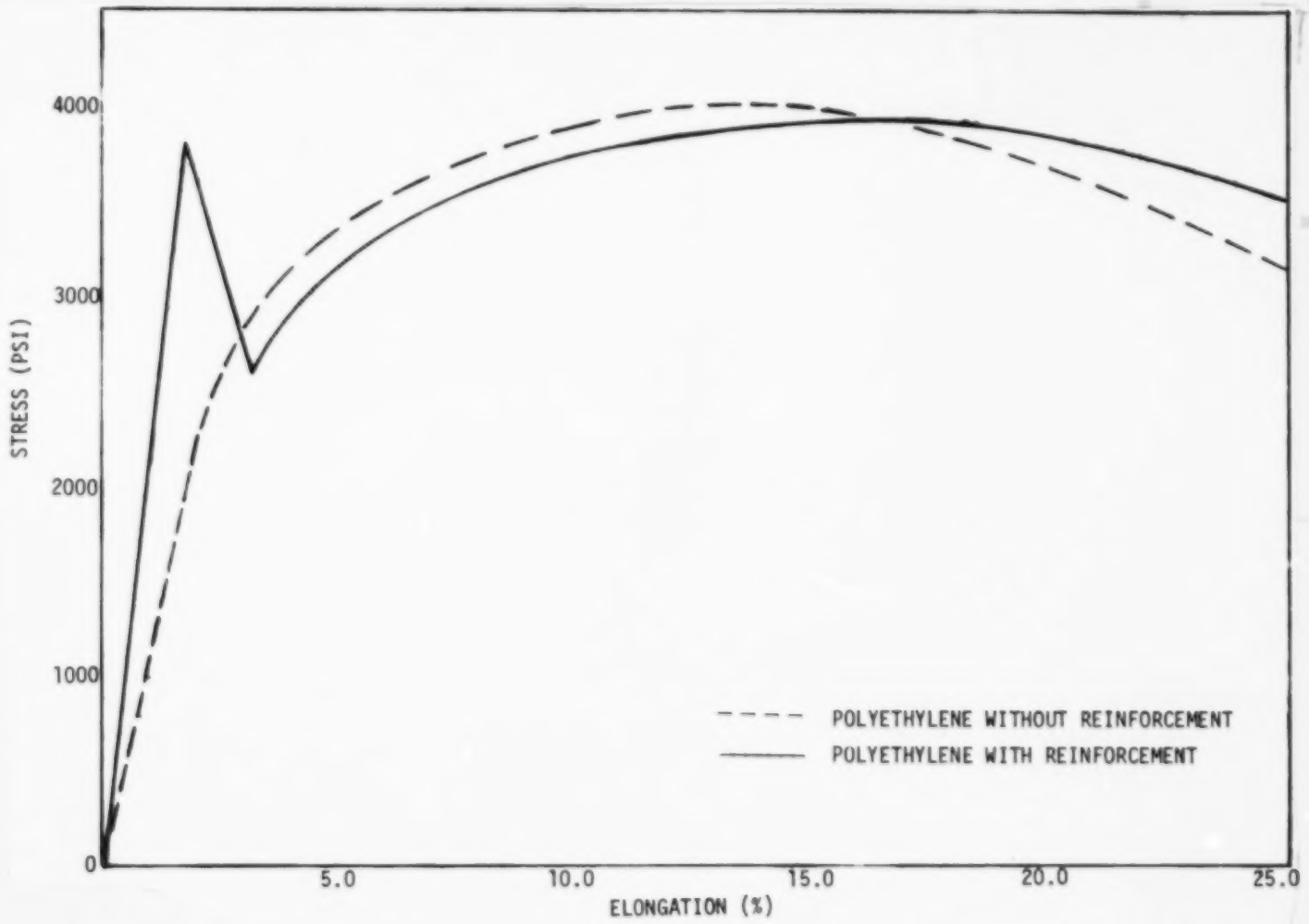

Figure 18. Comparison of stress-strain graphs for reinforced and unreinforced polyethylene.

TABLE 3. COMPARISON OF PROPERTIES OF SPECIMENS WITH AND WITHOUT REINFORCEMENT WITH VENDOR'S DATA

Property	Composite of PE + Reinforcement*	PE Alone*	Vendor
Tensile Strength @ Yield of PE	24.3 ± 4.2 MPa (3520 ± 610 psi)	26.8 ± 2.4 MPa (3890 ± 350 psi)	26.9 MPa (3900 psi)
Elongation @ Break	610 ± 100%	680 ± 120%	>650%
Elastic Modulus @ Origin	745 ± 138 MPa (108,000 ± 20,000 psi)	930 ± 210 MPa (135,000 ± 30,000 psi)	897-931 MPa (130,000-135,000 psi)

*Values are arithmetic means of five samples with their respective standard deviations, References 1 and 2.

TABLE 4. ADDITIONAL VENDOR'S DATA

Nominal Physical Properties of Marlex BMN TR-955

Property*	ASTM	English Units	English Value	Metric Units	Metric Value
Density	D1505	lbs/ft^3	59.6	g/cc	0.955
Melt Index	D1238	g/10 min.	6.5	g/10 min.	6.5
Flow Index (CIL), 375°F/190°C 1500 psi/10.4 MPa**	-	g/10 min.	7.4	g/10 min.	7.4
ESCR, Condition A, F_{50}	D1693	h	5-15	h	5-15
Brittleness Temperature	D746	°F	-180	°C	-118
Flexural Modulus	D790	psi	195,000	MPa	1345
Mesh Size	-		35		35
Tensile Strength @ Break	D638	psi	2150	MPa	14.8**
Impact Strength (notch method)	D256	FT-lb/in	1.75	J/m	93.3**
Hardness, Shore D	D676	-	60	-	60**
Vicat Softening Temperature	-	°F	259	°C	126**
Mold Shrinkage		in/in	.025-.045	mm	.635-1.14**

*Physical properties reported here were determined on compression-molded specimens prepared according to procedure C of ASTM D1928.
**Data obtained using a gas-operated extrusion plastometer based on a design by Canadian Industries, Ltd., with a die having an orifice diameter of 0.01925"/0.49mm and a land length of 0.276"/4.48mm.
***Reference 3.

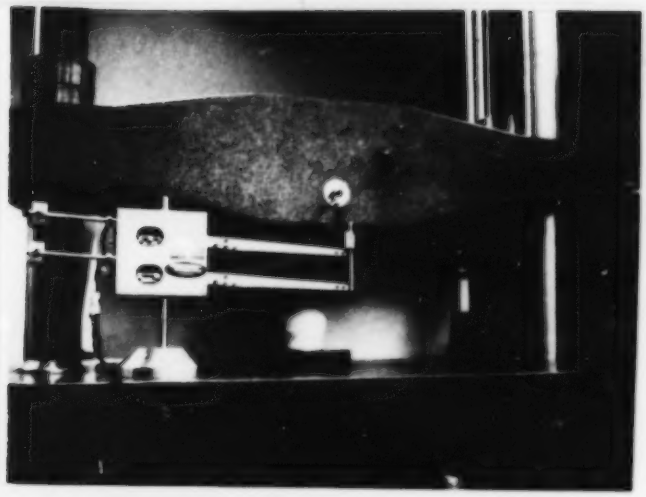

Figure 19. Onset of bulging of encapsulate under compression stress.

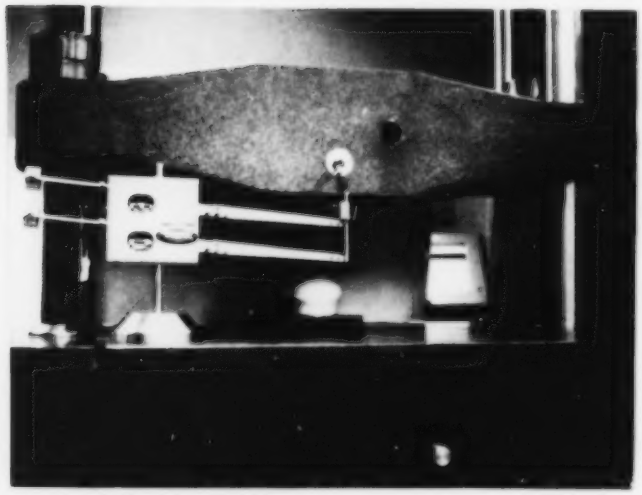

Figure 20. Pronounced bulging at specimen middle.

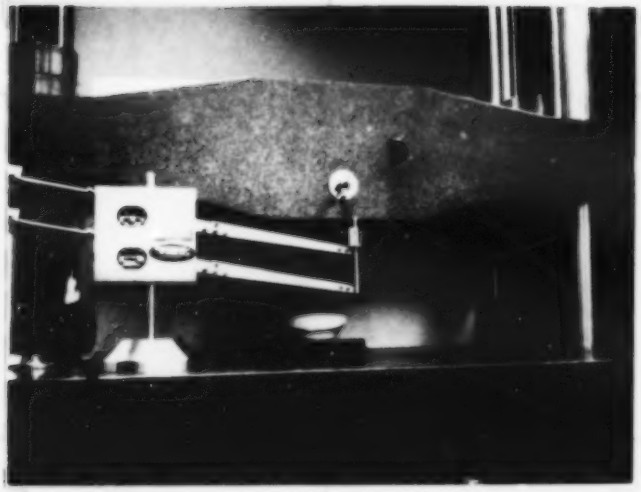

Figure 21. Further bulging.

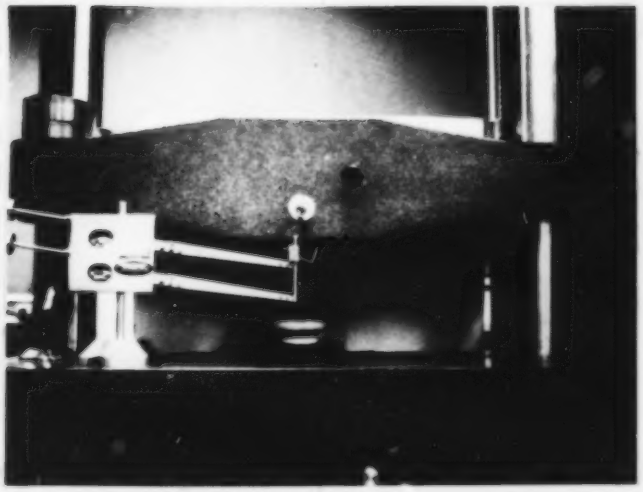

Figure 22. Vase-like configuration.

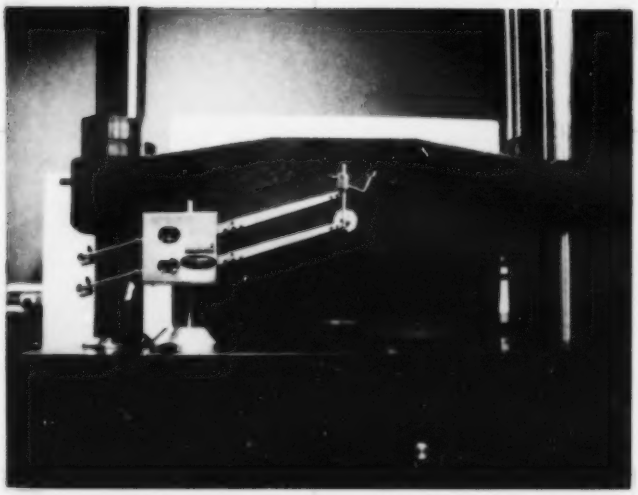

Figure 23. Encapsulate under extreme compression.

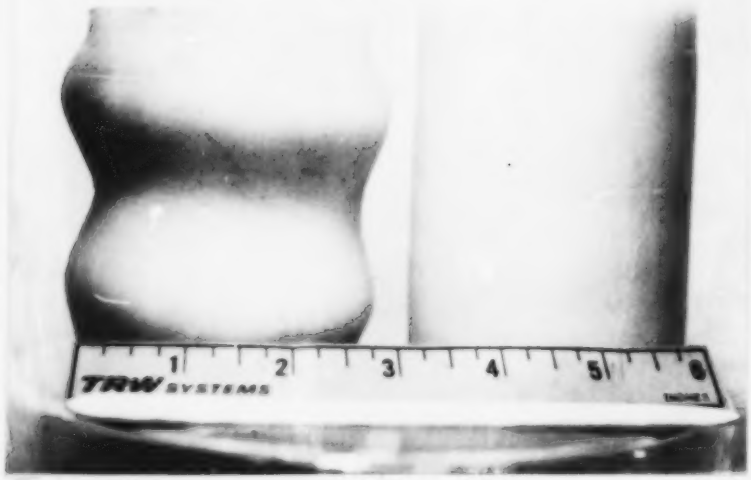

Figure 24. Shape of hollow encapsulate resulting from compression.

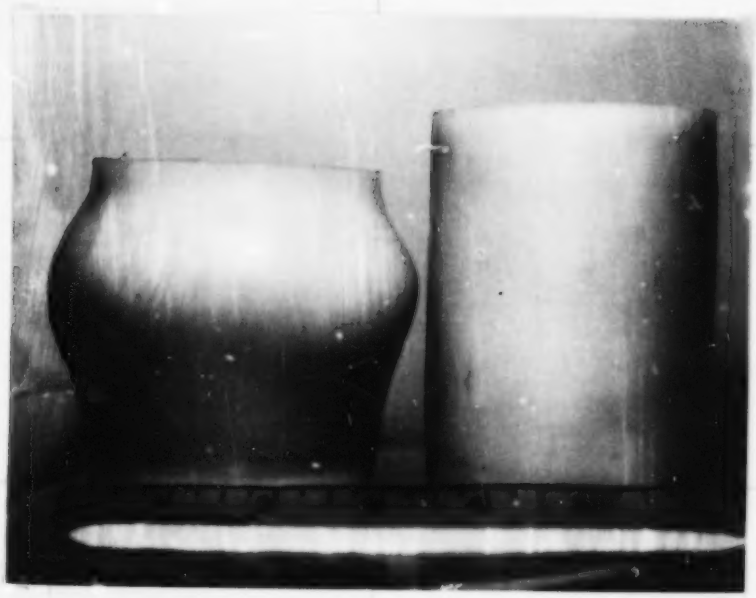

Figure 25. Shape of filled encapsulate resulting from compression.

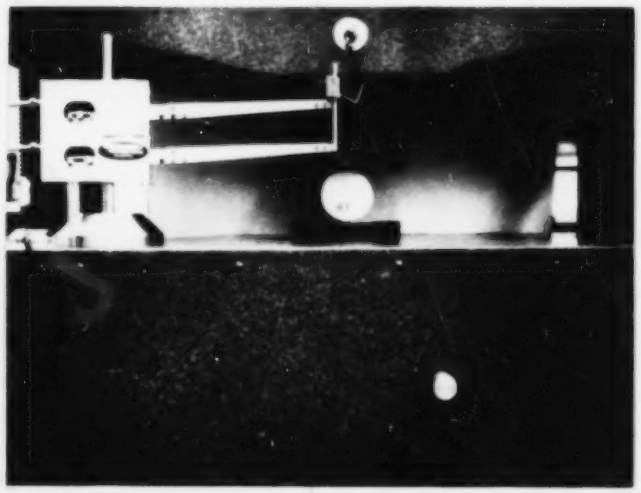

Figure 26. Encapsulate positioned for lateral compression.

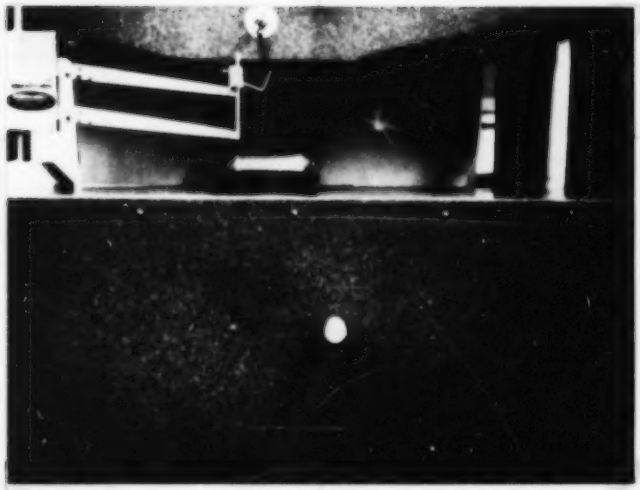

Figure 27. Side view of encapsulate during lateral compression.

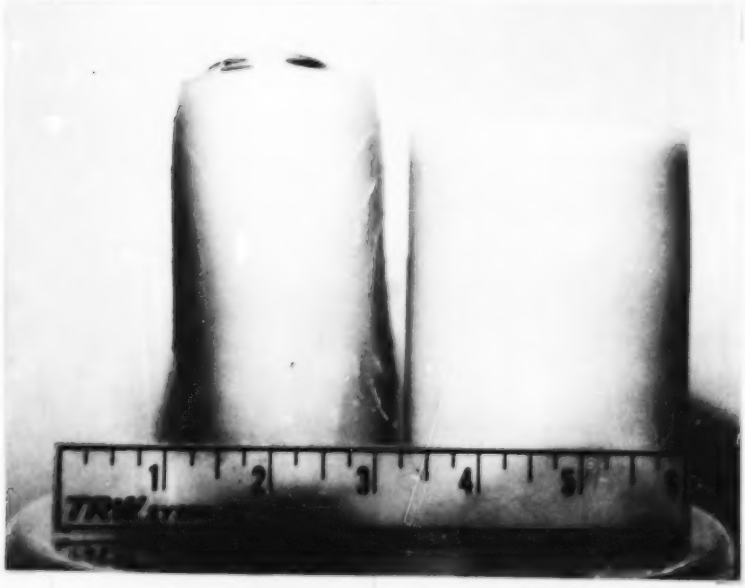

Figure 28. Side view of encapsulate after lateral compression.

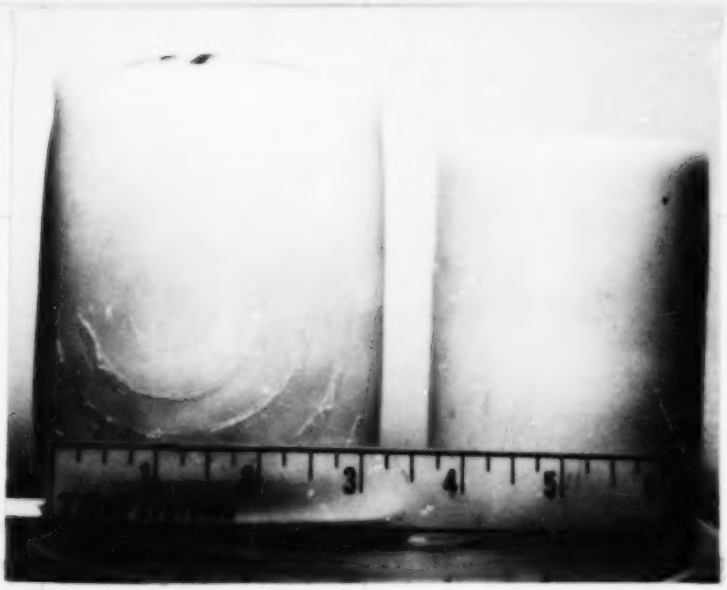

Figure 29. Front view of encapsulate after lateral compression.

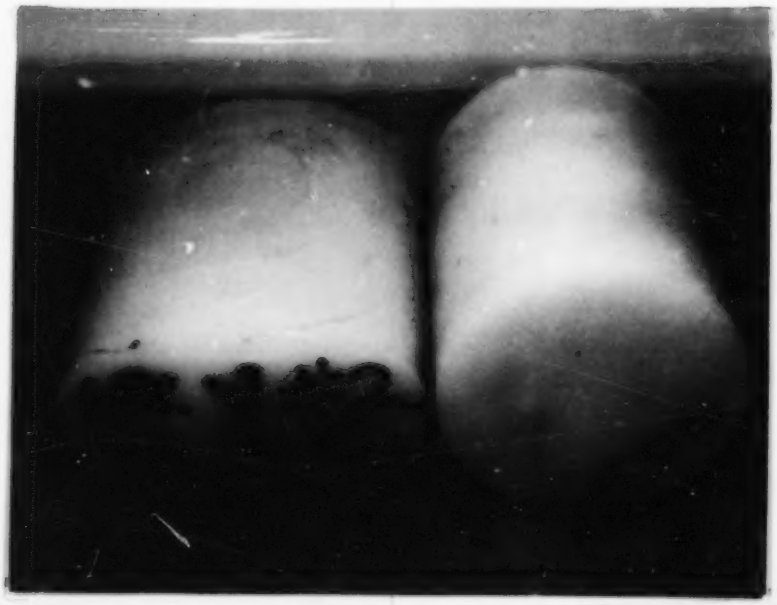

Figure 30. Comparative view of compressed and unperturbed specimen.

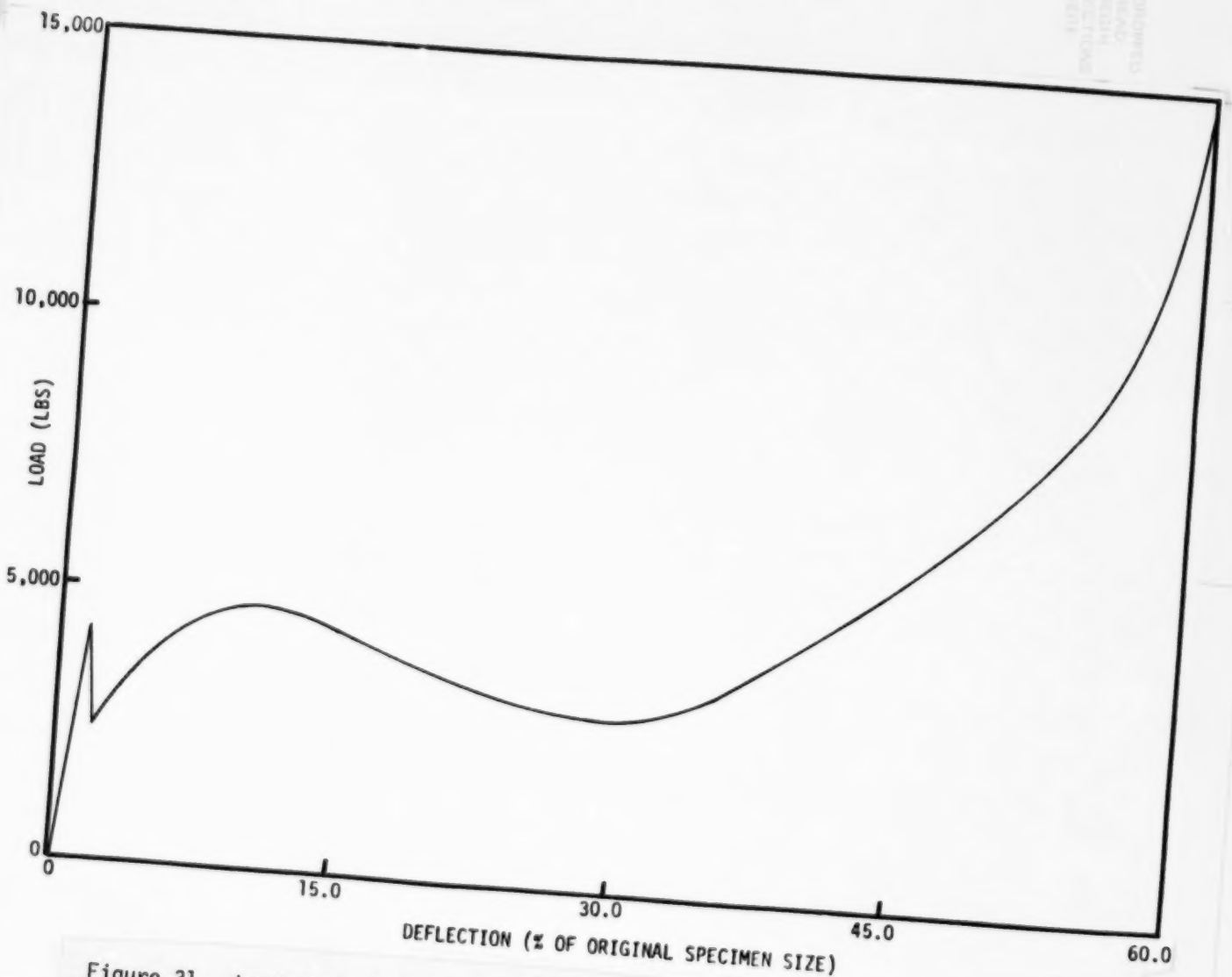

Figure 31. Load-deflection curve of hollow encapsulate (5,000- to 15,000-lb load).

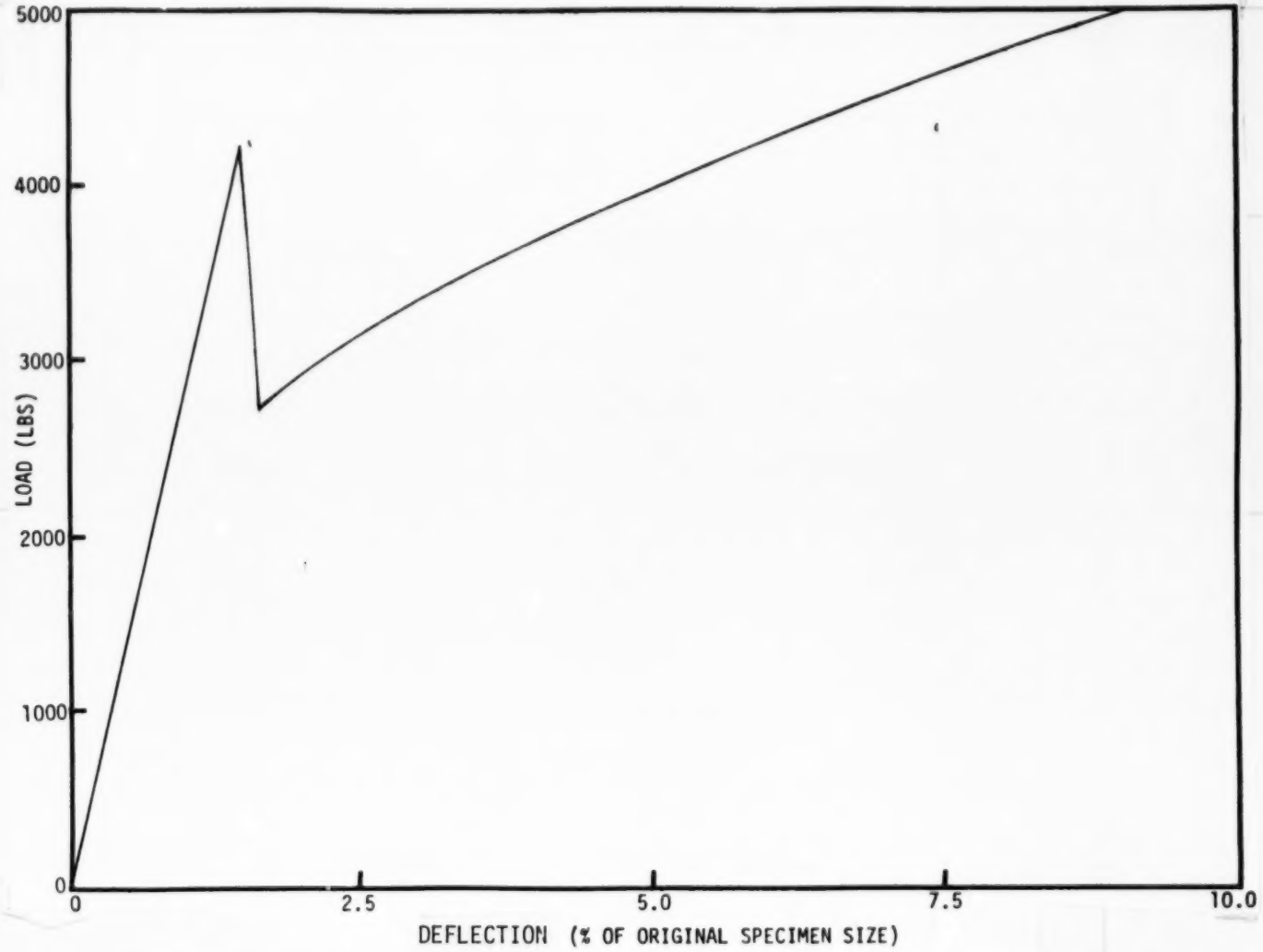

Figure 32. Load-deflection curve of hollow encapsulate (1,000- to 5,000-lb load).

the presence of the flexible polyethylene jacket. The remaining portion of the curve reflected the influence of the jacket. Up to 30% deflection the curve was characteristic of polyethylene in tension (see Figure 17). This was most likely a result of subjecting the jacket to tensile stresses after the casing underwent break in compression. After 30% compression, the apparent load-bearing properties of hollow encapsulates increased dramatically. This may be due to compression of the air in the encapsulates.

A view of the cross-section of a hollow encapsulate after compression to about 40% of its original vertical height is given in Figure 33. One notices the high degree of fragmentation of the casing. The fragments stemmed from the vertical inner walls, and the casing remained essentially intact on the top and bottom of the cavity. Almost all the casing was sheared from the jacket upon extreme compression of the encapsulate.

Data concerning hollow encapsulates under compression are provided in Table 5. Of particular interest was the percent recovery of encapsulates after compression to 40% of their original size. The percent recovery value was found to be about 90%. This high recovery, in our opinion, was due to minimal elongation of the polyethylene jacket in tension when the encapsulate was appreciably compressed. The amount of strain the jacket undergoes, as a result, may not have exceeded its elastic limit, thus the high recovery of original encapsulate dimensions. This phenomenon was

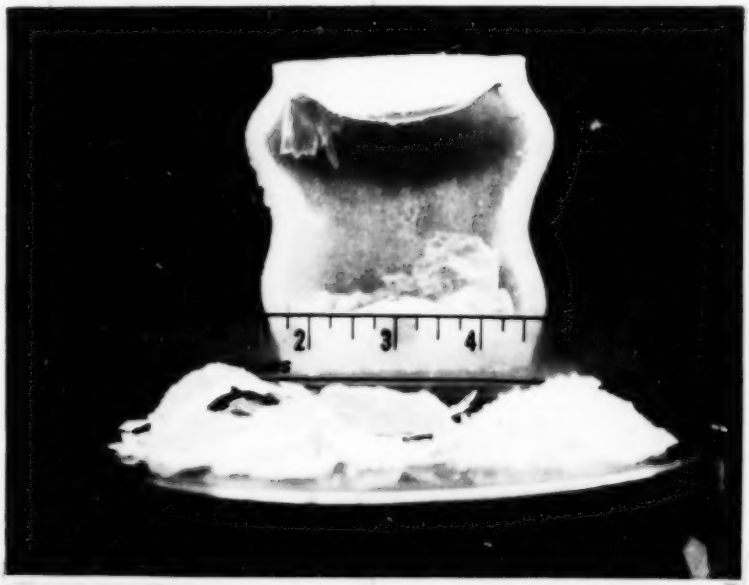

Figure 33. Fragmentation of the casing and functional integrity of the jacket after extreme compression.

TABLE 5. COMPRESSIVE PROPERTIES FOR HOLLOW SPECIMEN*

	International Units		English Units	
	Value	Unit	Value	Unit
Compression @ 1st Yield				
(based on total area)	4.16	MPa	603	psi
(based on wall area)	5.83	MPa	845	psi
Deflection @ 1st Yield	1.5	%	1.5	%
Transfer Stress from 1st Yield				
(based on total area)	2.35	MPa	341	psi
(based on wall area)	3.30	MPa	479	psi
Deflection @ Transfer	1.8	%	1.8	%
Compression @ 2nd Yield				
(based on total area)	4.72	MPa	684	psi
(based on wall area)	6.62	MPa	960	psi
Deflection @ 2nd Yield	10.5	%	10.5	%
Plastic Tenacity				
(based on total area)	3.07	MPa	445	psi
(based on wall area)	4.31	MPa	624	psi
Deflection @ Plastic Tenacity	29	%	29	%
Compressive Strength @ Maximum Compression				
(based on total area)	15.4	MPa	2230	psi
(based on wall area)	21.6	MPa	3130	psi
Maximum Deflection				
(based on original size)	40.8	%	40.8	%
% Recovery After Compression	89.6	%	89.8	%
Observations	No Observed Failure			

*Specimen Specifications

Total cross-sectional area = 6.97 in^2 or 4498mm^2

Cross-sectional area of walls = (4.97 in^2) or 3090mm^2

facilitated, in our opinion, by distribution of stress concentration on the casing through the medium of the jacket. Thereby the casing failed in compression by multiple cracking (see Figure 33) rather than by macrocracking of the casing. Distribution of stress on the plastic jacket by multiple cracking of the casing over a wide area caused minimal amounts of localized plastic strain in contrast to appreciable strain that would result from a few cracks.

When the encapsulates filled with sand were compressed, the spike nature of their compressive curves was essentially ameliorated. On Figure 34, the curve shows discontinuity at about 2.3% extension rather than at 1.5%, the position of the spike for hollow specimens. Further comparisons of hollow and filled specimens are given in Table 6.

An inner view is given in Figure 35 of an encapsulate that contained said filler that underwent extreme vertical compression. In this case, the casing of the filled encapsulate was not fragmented to the extent seen for the hollow one. One notices the casing to be appreciably rendered. Nevertheless, the jackets remained intact; there were no apparent breaks, nor were there any significant changes in their cross-sectional dimensions.

An inner view is given in Figure 36 of an encapsulate that contained hollow-sphere filler that underwent extreme lateral compression. In these cases, appreciable fragmentation of the casing occurred at the vertical extremities, whereas the casing disposed laterally was relatively unaffected. One can see the crease in the casing traversing the center caused by the extreme lateral compression. In lateral compression, as well as in vertical compression, the jackets of the encapsulates retain their functional integrity.

Mechanical Evaluations of Encapsulates

To produce suitably prepared encapsulates, it was necessary for the polyethylene to completely wet out the casings. Wet out required appropriate surface energetics of the adhesive (the plastic jacket) and the adherent (the casing). Furthermore, it required appropriate fluidity in the adhesive to effect during the processing time allotted a complete wet out of the adherent. In this respect, the surface temperature at the adherent was that of the adhesive. The adhesive did not degrade during processing, and thus upon consolidation, it gave rise to properties expected of the material.

LEACHING TESTS

The purpose of leaching tests was to examine the performance capabilities of fiberglass-reinforced polyethylene encapsulates holding heavy metals to withstand delocalization by aqueous and acidic solutions. Consequently, a broad spectrum of heavy metals representing commonly encountered industrial hazardous wastes were encapsulated. Each encapsulate was charged with a metal in solution and submerged in a 1500-ml leaching bath; samples of the baths were periodically withdrawn for chemical analysis. This work, described in detail below, indicated that encapsulation essentially pre-

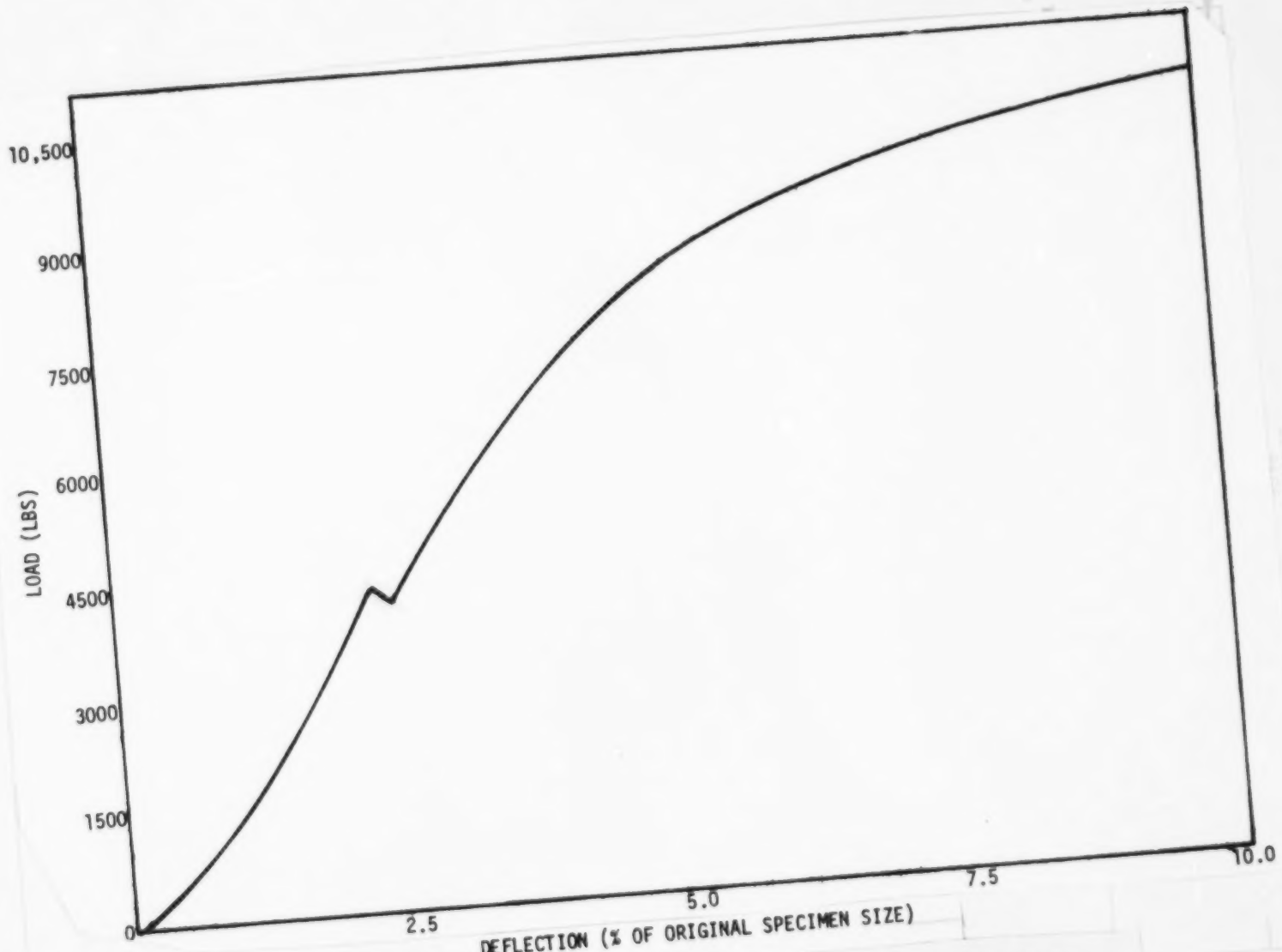

Figure 34. Load-deflection curve of sand-filled encapsulate.

TABLE 6. COMPRESSION DATA FOR COMPARISON OF HOLLOW AND SAND-FILLED SPECIMENS

Property	Hollow		Sand-Filled	
	S.I. Units	English	S.I. Units	English
Reinforcement				
Yield	4.16 MPa	603 psi	4.05 MPa	588 psi
(based on total area)	5.83 MPa	845 psi	-	-
(based on wall area)				
Deflection @ Reinforcement Yield	1.5%	1.5%	2.3%	2.3%
Stress Transfer upon Reinforcement				
Yield	2.35 MPa	341 psi	3.38 Ma	490 psi
(based on total area)	3.30 MPa	479 psi	-	-
(based on wall area)				
Deflection @ Stress Transfer	1.8%	1.8%	2.5%	2.5%

Figure 35. Inner view of encapsulate after extreme vertical compression.

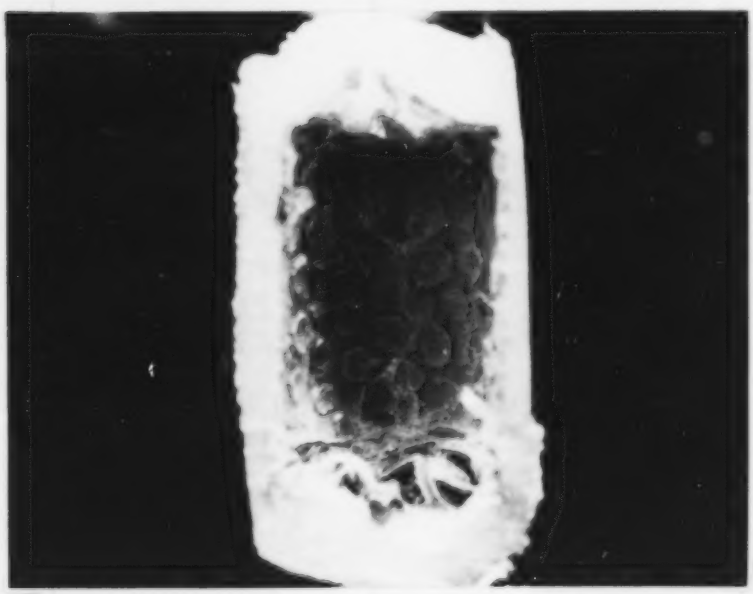

Figure 36. Inner view of encapsulate after extreme lateral compression.

cludes heavy metal delocalization during leaching by deionized water and acidic solutions.

Specimen Preparation

Saturated heavy metal salt solutions containing arsenic, cadmium, chromium, copper, mercury, nickel, lead, antimony, zinc and selenium were prepared by dissolving J.T. Baker reagent grade compounds (AS_2O_3, Cro_3, $CuCl_2$, $Hg(CH_3COO)_2$, $Ni(NO_3)_2 \cdot 6H_2O$, $Pb(CH_3COO)_2 \cdot 3H_2O$, $SbCl_3$, $ZnSO_4 \cdot 7H_2O$, SeO_2, respectively) in deionized water. Solution pH values were adjusted, when appropriate, to obtain maximum heavy metal solubility. A 25-ml aliquot of each solution was injected into different specimens as described in Section 5. Upon encapsulation, each specimen was washed in deionized water and submerged in a polyethylene screw-top container holding 1500 ml of deionized water. Encapsulates holding nickel, zinc and antimony were also submerged in 1500 ml of 0.1N HCl leaching solution.

Sample Preparation

Samples of leaching solutions were periodically withdrawn from each container with disposable-tip micropipets, as only a few microliters (10 to 25 μl) were required for a single analysis. The exception was with the As, Se and Hg determinations when a volume of 25 ml was necessary. Twenty-five milliliter aliquots were removed from these containers using an acid-washed glass pipet. The concentration effects involved with withdrawing samples were considered when calculating parts per million levels of cations during the 8-month leaching period.

Testing Method

The Jarrell-Ash 810 Atomic Absorption Spectrophotometer was used for all cation determinations with the exception of mercury. Cold vapor mercury determinations were conducted using an LDC UV monitor, Model 1205, and a standard stannous chloride reduction method.

At each sampling interval, aliquots of leaching baths were obtained and pH adjustments were performed using nitric acid, when necessary, to facilitate atomic absorption analysis. Samples were analyzed using the parameters listed in Table 7. For the graphite furnace techniques, a Jarrell-Ash FLA-10 graphite furnace attachment was utilized as the atomization source. In the cation analysis, using the hydride generation technique, a standard generator was used in conjunction with a standard Jarrell-Ash burner and mixing chamber. Additional discussion concerning atomic absorption parameters is provided in Appendix D.

Test Results

Tables 8 and 9 give heavy metal concentrations that were analytically detected in deionized water and 0.1N HCl leaching solutions, respectively. For each heavy metal, a set of standard solutions was prepared. Cation concentrations were derived from calibration curves generated for each set. Standard and specimen solutions were of similar background matrices.

The tables give the mean cation concentrations in parts per million

TABLE 7. ATOMIC ABSORPTION PARAMETERS

Element	Slit (Å) Widtn	Analytical Wave Length (nm)	Background Wave Length (nm)	Atomization Source	Detection Limit (ppm)	Method/Parameters
As	10	193.7	Continuum	Ar/H_2 Flame	0.001	Catalytic conversion of antimony to stibine then burning gas in Ar/H_2 flame.
Cd	4	228.8	226.5	Graphite Furnace	0.0006	Dry 30 sec. @ 100°C Ash 30 sec. @ 250°C Atomize 5 sec @ 2200°C Ar 4L/min. 10 µl sample plus 10 µl 0.2% Ammonium Molydate
Cr	4	357.9	351.9	Graphite Furnace	0.0005	Dry 30 sec @ 200°C Ash 30 sec @ 1000°C Atomize 7 sec @ 2800°C 25 µl sample Ar 2 L/min.
Cu	10A	324.7	323.4	Graphite Furnace	0.001	Dry 30 sec @ 200°C Ash 30 sec @ 900°C Atomize 6 sec @ 2800°C 25 µl sample Ar 3 L/min
Hg	UV Monitor	254.0	----	Glass Frite	0.0003	Stannous chloride reduction
Ni	2	232.0	231.6	Graphite Furnace	0.003	Dry 40 sec @ 150°C Ash 30 sec @ 800°C Atomize 10 sec @ 2700°C 25 µl sample Ar 3 L/min
Pb	10	2170	----	Graphite Furnace	0.002	Dry 40 sec @ 100°C Ash 60 sec @ 600°C Atomize 7 sec @ 2000°C Ar 4 L/min 10 µl sample plus 10 µl 0.2% Ammonium Molybdate 1.0% HNO_2
Sb	10	2176	----	Ar/H_2 Flame	0.002	Catalytic conversion of antimony to stibine then burning gas in Ar/H_2 flame.
Se	10	1960	1942	Graphite Furnace	0.01	Dry 40 sec @ 150°C Ash 30 sec 600°C 10 µl sample plus 10 µl 0.2% HNO_3 100 ppm Ni (as $NiNO_3$)
Zn	10	2139	2204	C_2H_2/air Flame	0.003	Direct atomization of sample.

TABLE 8. METAL CONCENTRATIONS IN DEIONIZED WATER LEACHING SOLUTION

(ppm)

Metal	Day* 90	Day 120	Day 240	Metal Loss** (Calculated)	Comments
As	-	< 0.002 (±0.001)	0.002 ± 0.002	1000	
Cd	0.05	< 0.0006 (±0.0006)	0.0005 ± 0.0005	800	Refinement of detection limits due to utilization improved atomization sources in Atomic Absorption analysis.
Cr	0.05	< 0.007 (±0.0005)	0.004 ± 0.001	810	" " "
Cu	0.05	< 0.009 (±0.001)	0.009 ± 0.001	3190	" " "
Hg	2	< 0.002 (±0.0003)	0.0026 ± 0.0003	820	" " "
Ni	0.5	< 0.007 (±0.003)	0.013 ± 0.002	810	" " "
Pb	1.0	< 0.003 (±0.002)	0.007 ± 0.002	1000	" " "
Sb	1.0	< 0.040 (±0.002)	0.023 ± 0.002	860	" " "
Zn	1.0	< 0.003 (±0.003)	0.003	2070	" " "
Se	-	< 0.117 (±0.011)	0.010 + 0.011	860	" " "

*"Day" - Number of days encapsulated waste immersed in deionized water.

**"Metal Loss" - Values calculated give concentrations of contaminants in leaching solutions assuming catastrophic failure of specimens

TABLE 9. METAL CONCENTRATION IN 0.1N HCl LEACHING SOLUTION

(ppm)

Metal	Day* 60	Day 90	Day 210	Metal Loss** (Calculated)	Comments
Ni	< 0.5	0.010 (±0.003)	0.005 ± 0.002	810	Refinement of detection limits due to utilization of improved atomization sources in Atomic Absorption analysis.
Sb	< 1.0	0.040 (±0.002)	0.026 ± 0.002	860	" " "
Zn	< 0.05	0.006 (±0.003)	0.058 ± 0.003	2070	" " "

*"Day" - Number of days encapsulated waste immersed in HCl leaching solution.

**"Metal Loss" - Values calculated give concentrations of contaminants in leaching solutions assuming catastrophic failure of specimens.

(ppm) and the standard deviation of four readings that were taken for each determination. The values listed under "Metal Loss" are the concentrations of metals that would be detected in the leaching baths if encapsulates failed catastrophically. These comparative data clearly demonstrate the effectiveness of encapsulation in localizing heavy metals when exposed to deionized water and acidic solutions.

SECTION 7

ENGINEERING SUBTASK

This section provides a conceptual view of a fabrication apparatus for making large fiberglass-casing-reinforced polyethylene containers. Described here is a commercial-scale mold for fusing a seamless polyethylene outer wall onto a glass fiber inner shell. The energy requirements are enumerated. The cost for encapsulation of a 55-gal drum was estimated at $30 per drum.

DESIGN OF THE POLYETHYLENE FUSION MOLDS

Though the equipment for spraying the fiberglass-reinforced resin are standard commercial items, no such equipment for fusing the polyethylene jacket to the inner shell was available. A preliminary design for this mold has been made and is shown in Figures 37 and 38. Figure 39 shows how two molds are connected with the heaters and coolers to supply and remove heat from the molds.

The mold consists of a pivotally mounted cylindrical mold. The ends or caps are formed by two hydraulic pistons mounted on the same yoke that supports the mold. The mold is of a clam shell design, which permits side loading of the shell. In operation, the clam shell is opened and the shell is inserted into the mold and positioned. The mold is closed, and sufficient powdered polyethylene to form the sides and top of shell are added. The hydraulic rams close the mold and heat is applied to the top and sides by flowing a hot heat transfer fluid such as Therminal 66 through heat coils. The maximum molding temperature is 500°F. It is desired to apply the heat as rapidly as possible and then cool the molten polyethylene to reduce the heat transfer through the inner shell to the barrel. Thus, the highest allowable temperatures should be used in the heat transfer fluid. After the sides and top have been fused and cooled to solidify, the mold is turned over, the upper hydraulic ram is opened and sufficient powdered polyethylene is added to form the lid. The upper hydraulic ram is now closed and heat is applied to the lid and edge of the mold at the line where the lid must fuse to the sides. After the lid has been fused and solidified, the mold is opened and the finished encapsulate is ejected.

The fusion mold has been designed to be energy conservative. By valving two molds together, it is possible to recover about 40% of the heat rejected from a hot mold during cooling to heat a cold mold during heating. This is shown in Figure 39. Here the major components are seen to be an oil-fired heater with an air preheater, an air-cooled radiator, two fusion molds and associated pumps and plumbing. The operation of the molds follows:

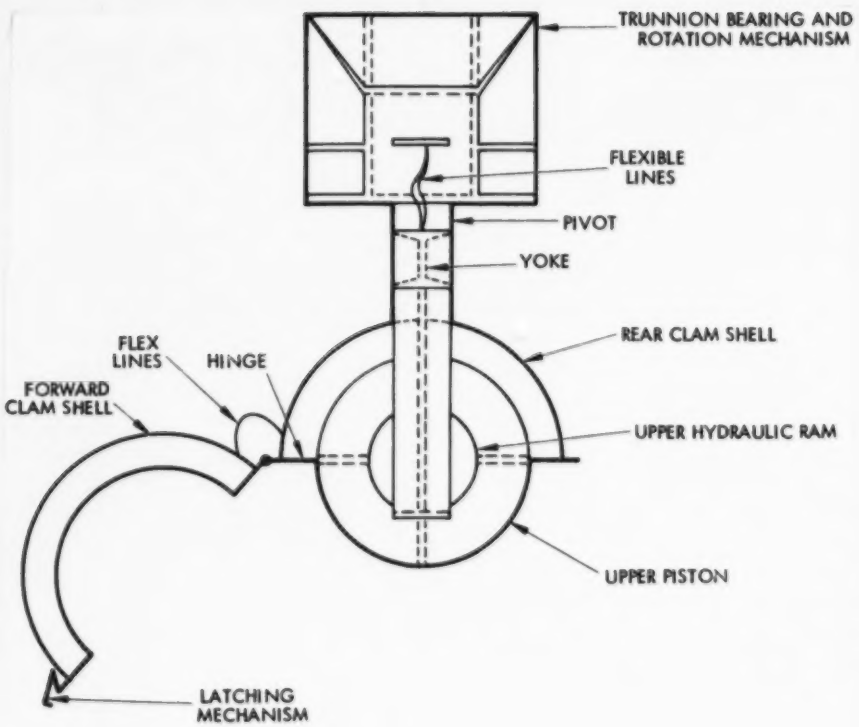

Figure 37. Top view of polyethylene fusion mold showing clam shell open.

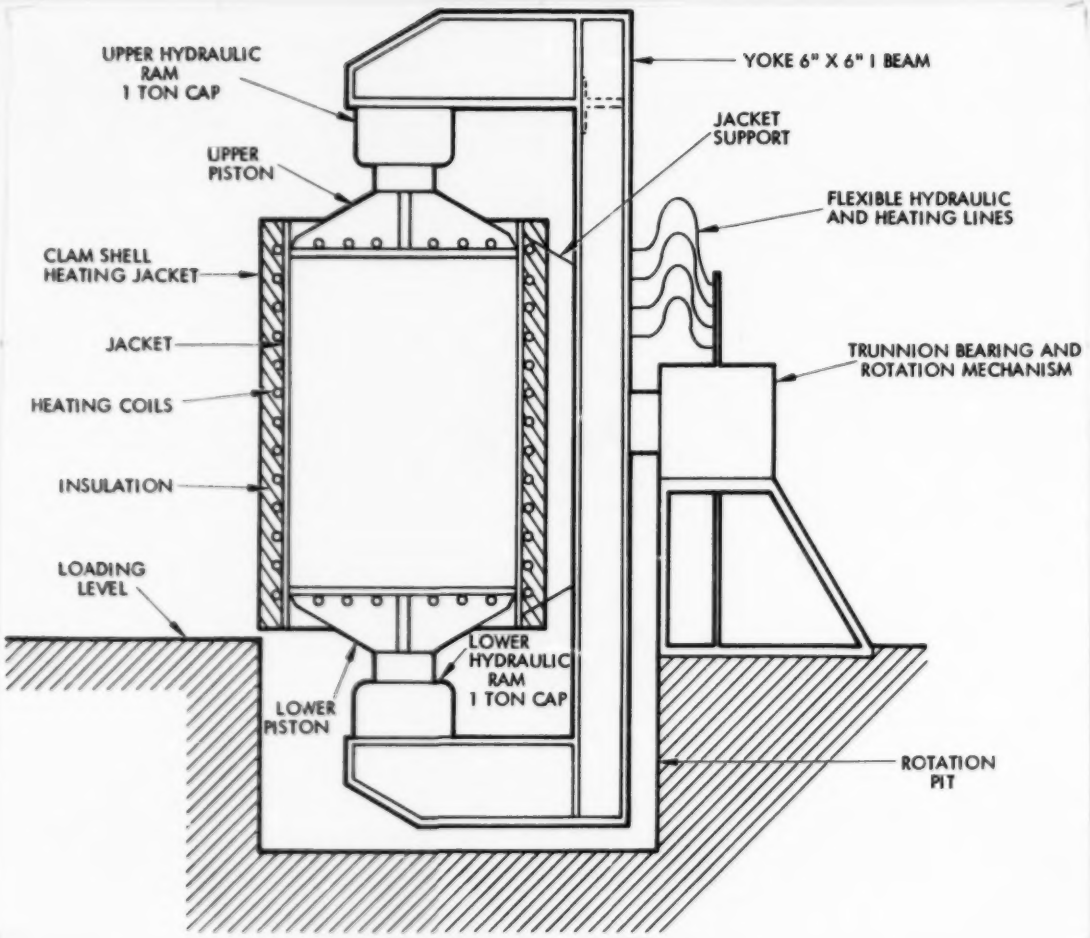

Figure 38. Side view of the polyethylene fusion mold.

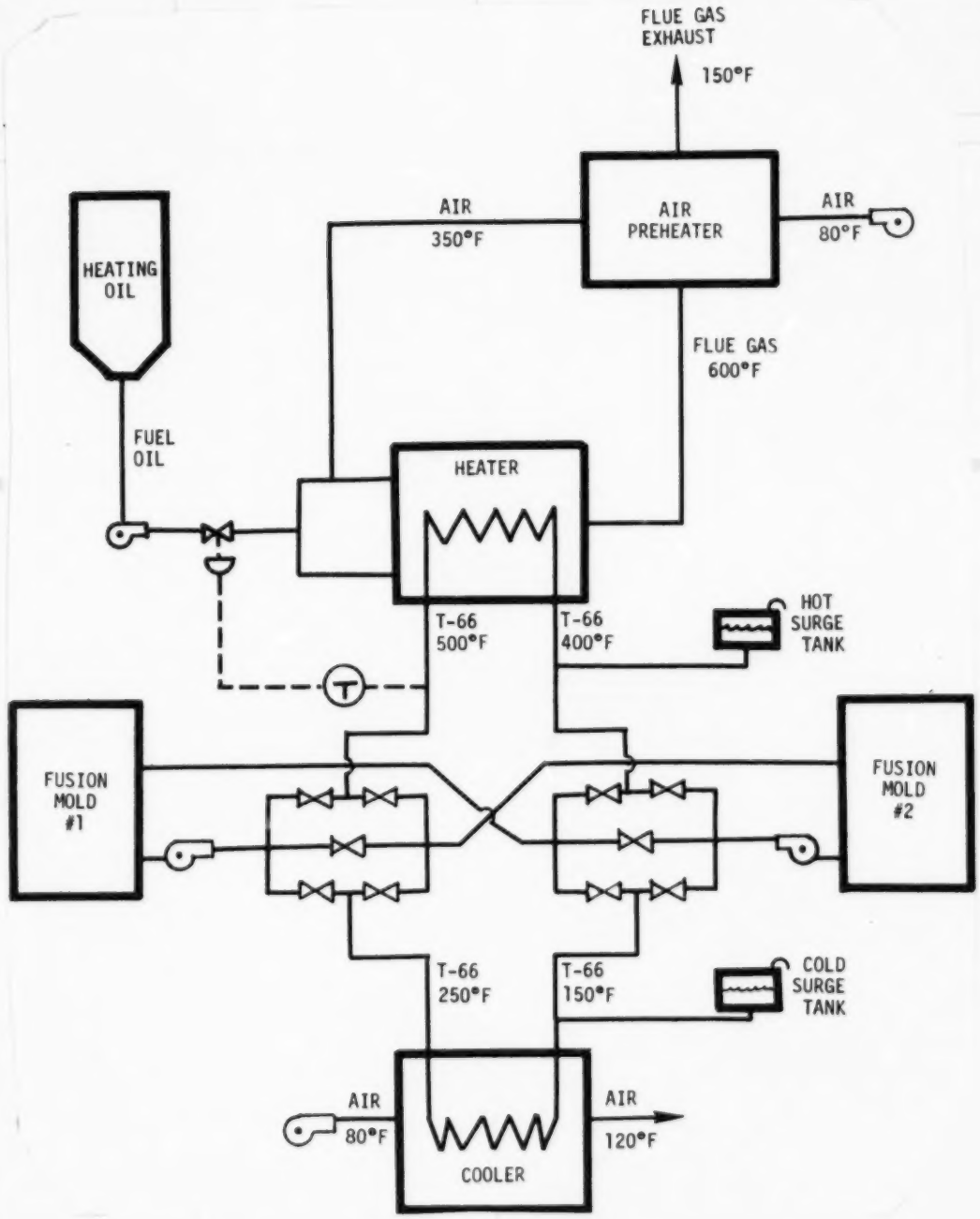

Figure 39. Piping diagram for the polyethylene fusion mold.

1. With one mold hot and the second cold, the heat transfer fluid recirculates between the molds cooling the hot mold from 450° to 300°F while the cold mold heats from 100° to 250°F.

2. The recirculation valves are closed and the molds connected to the heater and cooler where the hot mold is cooled from 300° to 150°F while the cold is heated to its maximum temperature of 450°F. The molds have now interchanged their thermal state.

3. The cooled encapsulate is removed, another added, and the sequence is repeated.

Figure 39 shows the position of the control valves for recirculation, heating and cooling. These valves may be electrically operated and their sequencing automatically controlled.

A comparison of the energy requirements for different mold designs is interesting. If the mold was electrically heated and all the heat rejected during cooling, then for every BTU delivered to the mold, 3 BTU would be required to generate the electricity. If the mold was heated with an 80% efficient furnace, then 1.25 BTU would be required for each BTU delivered. In the present design using recirculation approximately 40% of the heat is recovered; thus for each delivered BTU, the furnace must only supply 0.75 BTU; the balance comes from the recirculation between the molds. Thus the present design uses 25% of the energy that an electrical design would use.

ESTIMATED COST

In a more detailed design phase, one would expect to define the costs accurately. Without such data, however, the cost may be treated parametrically, as shown in Figure 40. Here the encapsulation costs in dollars per ton are plotted as a function of the density of the waste in the encapsulate. This is prepared for a 55-gal drum encapsulate. For example, for a waste with a density of 80 lb/ft^3 and an encapsulation cost of $30 per drum, the cost is $96/ton.

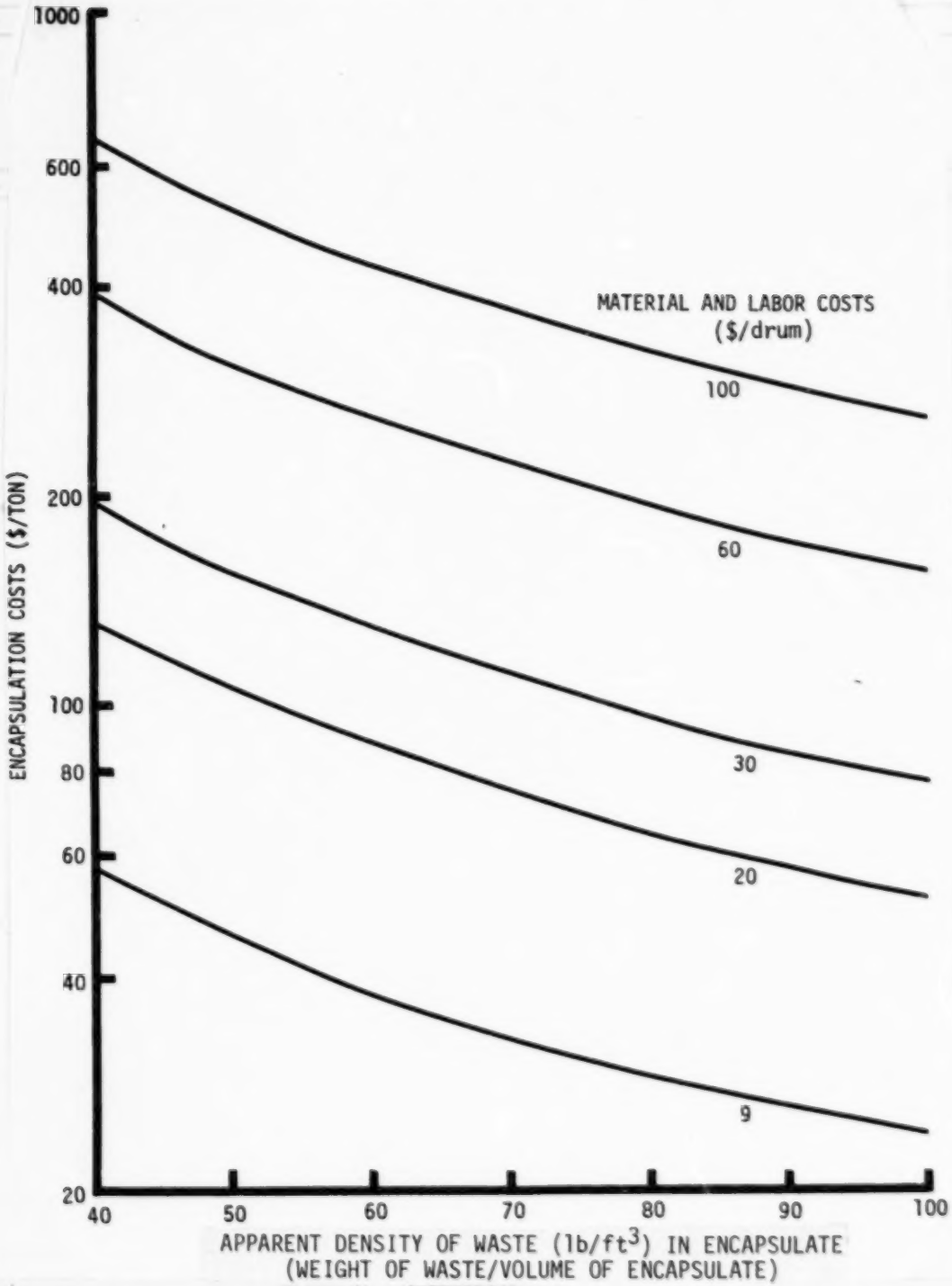

Figure 40. Encapsulation costs as a function of the density of the waste.

SECTION 8

RESULTS AND DISCUSSION

This work presents high-performance containers for encapsulating containerized hazardous wastes. They were characterized by watertight, homogeneous polyethylene plastic outer walls reinforced by fiberglass inner walls. The plastic of the outer walls exhibited the mechanical properties, toughness, and flexibility expected from polyethylene resin. No degradation of resin properties was found to occur in the outer wall fabrication process.

The evaluations indicated excellent performance capabilities for large containers. In the same vein, the high performance of 1/4-in.-thick commercial polyethylene 55-gal drums was well known. But even better performance was expected from commercial-scale containers according to this work because their walls would be seam-free and reinforced by fiberglass, features that did not characterize commercial 55-gal plastic drums.

The technique employed for fabricating containers was comparable to that employed in the fiberglass-reinforced plastic (FRP) industry. In the latter case, for making shower stalls, bath tubs, and plastic boats for example, a gel coat was sprayed onto appropriately shaped surfaces acting as molds and the coating was reinforced by spray-up of chopped fiberglass. One role of the gel coat was to seal the structure. In this work the polyethylene outer wall of the container was viewed as a gel coat. Whereas in FRP the gel coat was applied first followed by the fiberglass, here the fiberglass casing was fabricated first then the gel coat acted as the outer wall. The properties of the outer walls, however, differed significantly from state-of-the-art gel coats. The former, freestanding, as shown in this study were tough and flexible; whereas gel coats, unsupported, were brittle in nature.

The method of molding the polyethylene outer walls was comparable to that employed in the rotomolding industry. This industry fabricated large PE vessels for containing corrosive chemicals from particulated polyethylene in rotating and/or rocking molds. Here, in contrast, the mold was stationary, and in addition contained an insert.

The containers described here are new structural compositions in the plastics art. Review of the technical literature indicated that the containers were unique.

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APPENDIX A

POLYBUTADIENE-POLYETHYLENE SHEET

In the laboratory, a sheet was fabricated from polyethylene powder and a polybutadiene-urethane binder. The sheet was thermally fused under mechanical pressure into translucent plastic that exhibited exceptional stiffness. Figure A-1 shows the sheet and plastic.

From the viewpoint of realizing new plastic consistencies and materials, the accomplishment of making sheet capable of being readily draped and then being fused into stiff translucent plastic can be of significance, particularly in laminate formation. The plastic showed excellent stability to chemical reagents. However, draping the sheet over a container and then fusing the plastic thereon was not a tenable operation because the operation required higher mechanical pressures than those appropriate for securing wastes. Aside from reporting such materials as fall-out technology constituting invention or innovation, no further characterization was carried out. However, disclosure of invention was made for the purpose of drafting an application for patent on behalf of the Environmental Protection Agency. Further characterization studies were not made because contractual requirements limited work to those compositions with potential for ready processing into ensembles for securing preconfined hazardous wastes.

Unfortunately, various sheet formulations investigated required appreciable mechanical pressure for fabricating. The need for mechanical pressure introduced a steep escalation in sophistication of apparatus required for making encapsulates. Since it seemed unlikely that sheet can be fabricated with minimal mechanical pressures, the sheet approach was set aside for use in container encapsulation. Nevertheless, the sheet can be of interest in commercial applications because mechanical pressures employed in laminate fabrication, for example, are adequate for fabrication of sheet. The sheet invented in this program can, for example, be used to fashion the "top coating" of a laminate, thereby, yielding a surface with excellent chemical stability.

In future investigations of the polyethylene/polybutadiene systems, polyethylene powder was coated with polybutadiene and then fused. These resin systems were found to fuse under minimal mechanical pressures. The fusion of polyethylene/butadiene introduced desirable chemical cross-links in the resin.

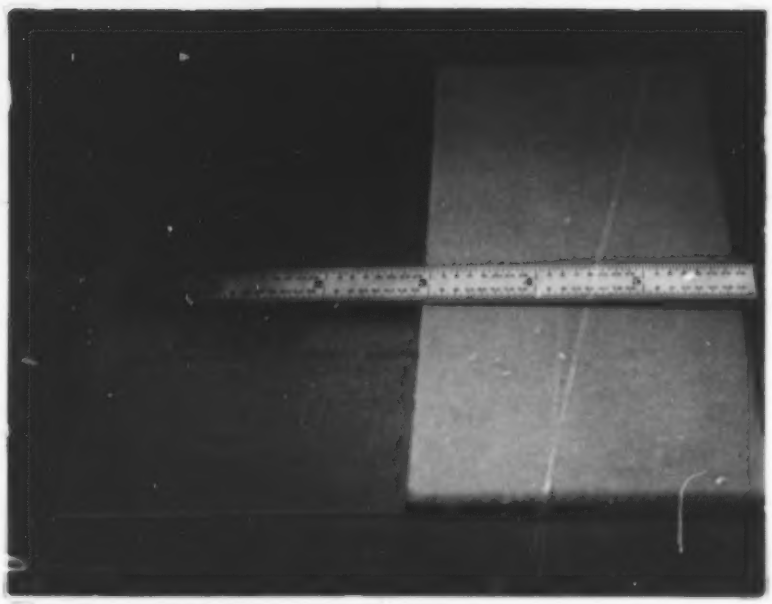

Figure A-1. Sheet of powdered polyethylene in polybutadiene-urethane binder (right) and thermally fused sheet (left).

APPENDIX B

ENCAPSULATION STUDIES

The laboratory work was geared to preparing test specimens simulating the envisioned field size ensembles of small containers. The specimens were evaluated according to specifications in the contractual requirements. It should be pointed out that appreciable performance data were available from the vendors concerning the polyethylene resin employed in fabricating test specimens. Since these data pertained to unreinforced resin, it was our expectation that enhanced performance would characterize resin jackets of this study due to the reinforcing fiberglass casing onto which they were fused.

A reinforcing casing was fashioned about an agglomerate of marbles simulating containers. Figure B-1 shows the resulting assemblage. Since spray-up can not be carried out conveniently in fabrication of laboratory-size test specimens, glass fabric rather than chopped glass was employed in making the reinforcement.

The agglomerate of Figure B-1 was fitted with a jacket of commercial polyethylene employing the mold shown in Figure B-2.

Figures B-3 and B-4 show the test specimen in position for the final jacketing step, which involves fusing the cover onto the prefashioned assemblage. Inside the assemblage, the spheres appear as white blotches, which simulate encased containers.

Two jacketed test specimens were photographed (Figures B-5 and B-6). The resin flash appears in Figure B-6 and not in Figure B-5. The absence or presence of visual flash depended on fabrication conditions. Since the covering resin was fused at the lips of the prefashioned object as shown in Figure B-4, the nature of the flash will not, in our opinion, have a bearing on product performance.

A test specimen was sectioned and the reinforcement was stripped (Figure B-7). Notice that the covering resin is homogeneously fused onto the lips of the prefashioned jacket. The casing was completely wetted out by the polyethylene, as expected, because of the high surface energy of the reinforcement and the low energy of polyethylene resin. Furthermore, the resin impregnated the casing and wetted out the inner side. This wet-out and impregnation phenomenon was very desirable, in our opinion, because it gave rise to optimal reinforcement of the jacket by the glass.

The demonstration of encapsulation was extended to thin-walled, hollow,

plastic microballoons; these materials simulate soft-walled containers. Figure B-8 shows the two types of simulated containers used in this work (i.e., marbles and plastic microballoons). A cross-section of an encapsulate holding the microballoons is shown in Figure B-9.

Preliminary work was carried out to show the feasibility of encapsulating a steel juice can containing liquid. This container simulated a 55-gal drum. Here the casing contained sand, which filled the annular space between the can and casing walls. This assemblage was then encapsulated by polyethylene.

Figures B-10 through B-12 are provided to show encapsulation of a container simulating a 55-gal drum. A satisfying aspect of this demonstration was the potential feasibility of general application of encapsulation. The procedure was found to be applicable to various kinds of containers.

Figure B-1. Agglomerate of spheres encased by glass-resin cocoon.

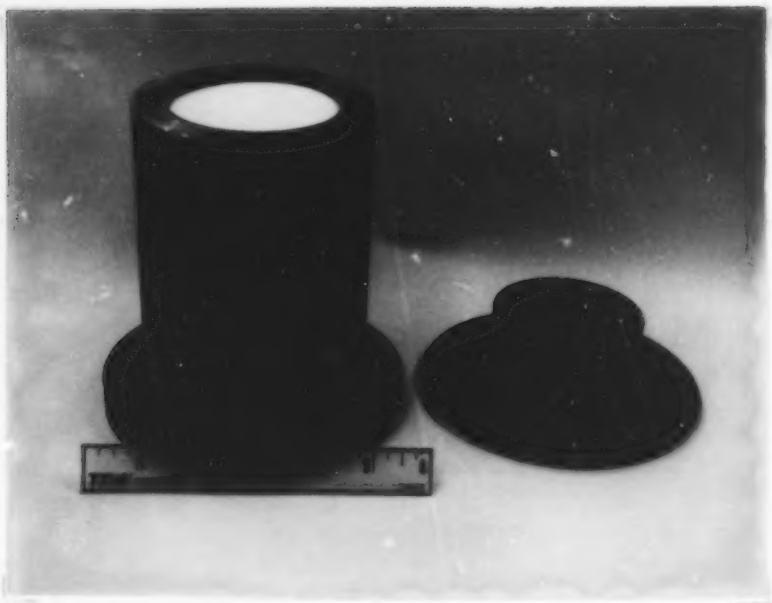

Figure B-2. Mold and test specimen covered by powdered polyethylene.

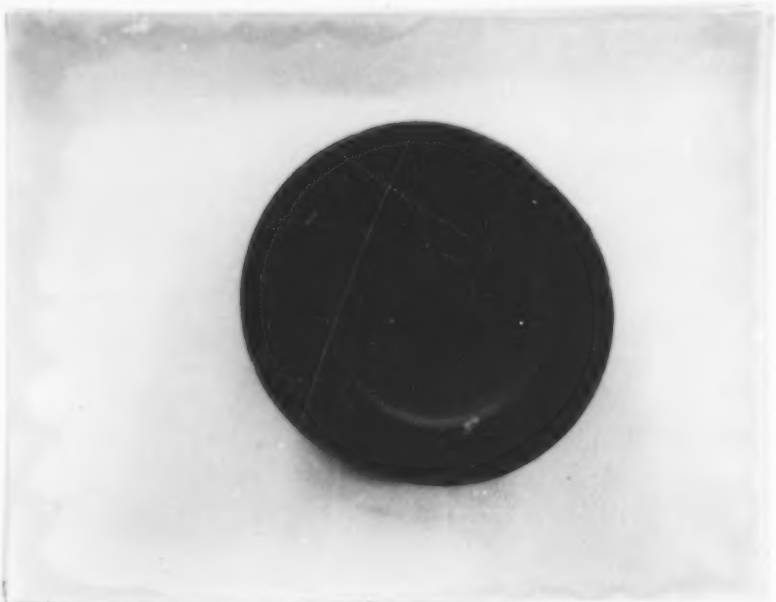

Figure B-3. Test specimen positioned for final jacketing step.

Figure B-4. Another view of test specimen positioned for final jacketing step.

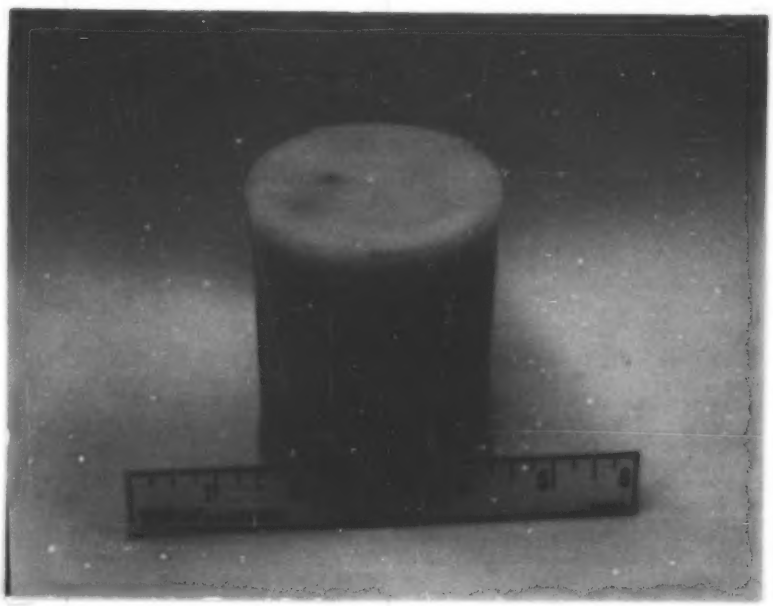

Figure B-5. Polyethylene-encapsulated test specimen.

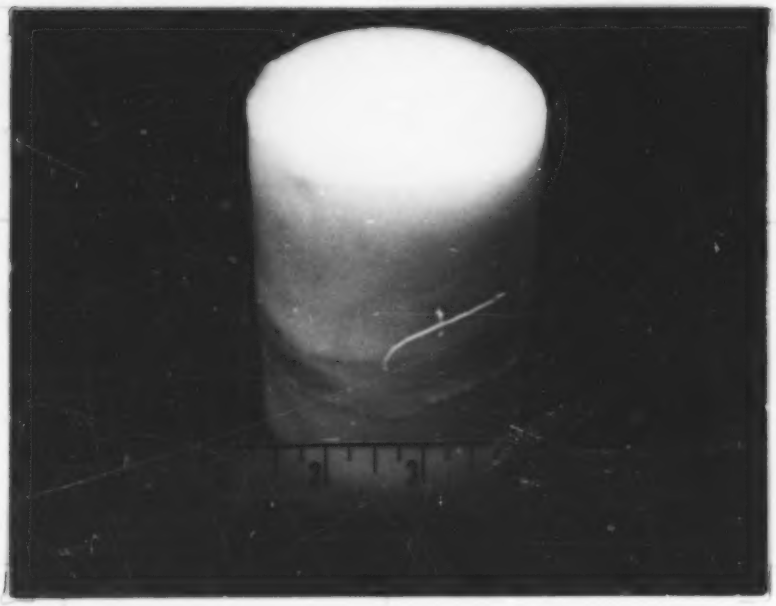

Figure B-6. Polyethylene-encapsulated test specimen showing resin flash.

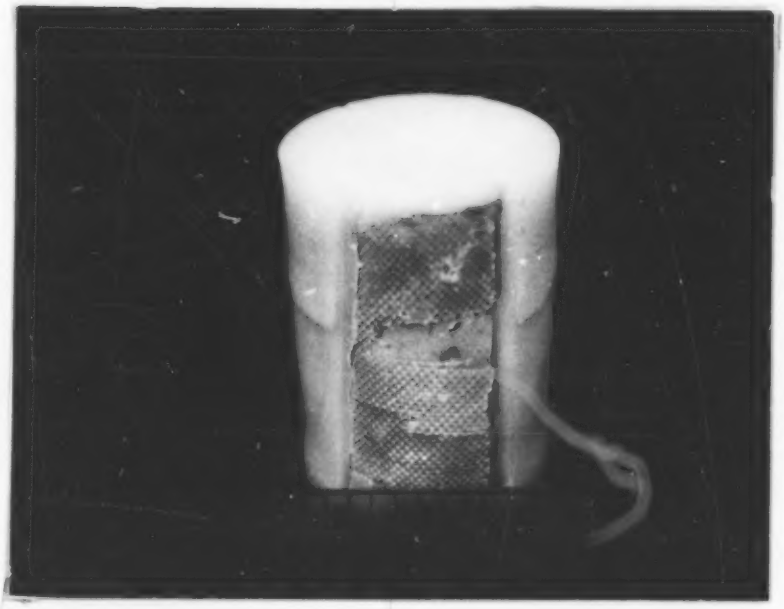

Figure B-7. Sectioned test specimen of encapsulated, simulated containers.

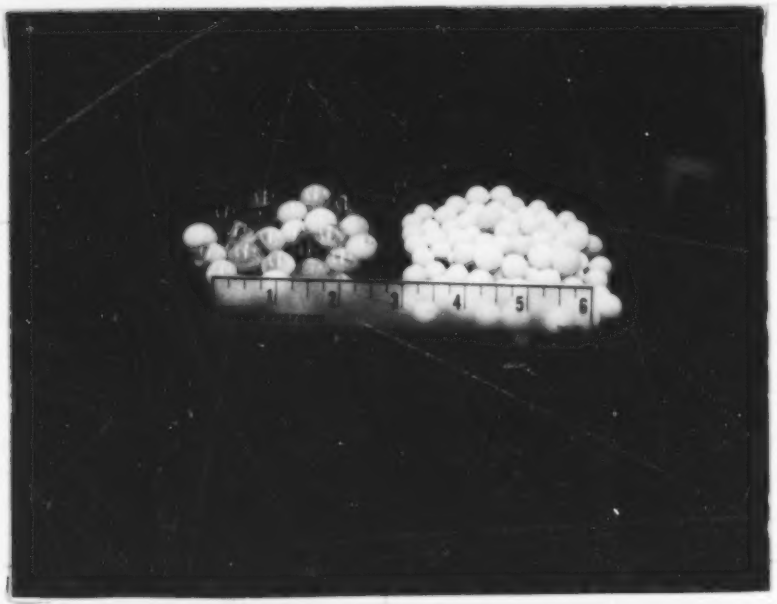

Figure B-8. Two types of spheres simulating containers employed in fabricating encapsulated specimens: Left, marbles; right, hollow plastic microballoons.

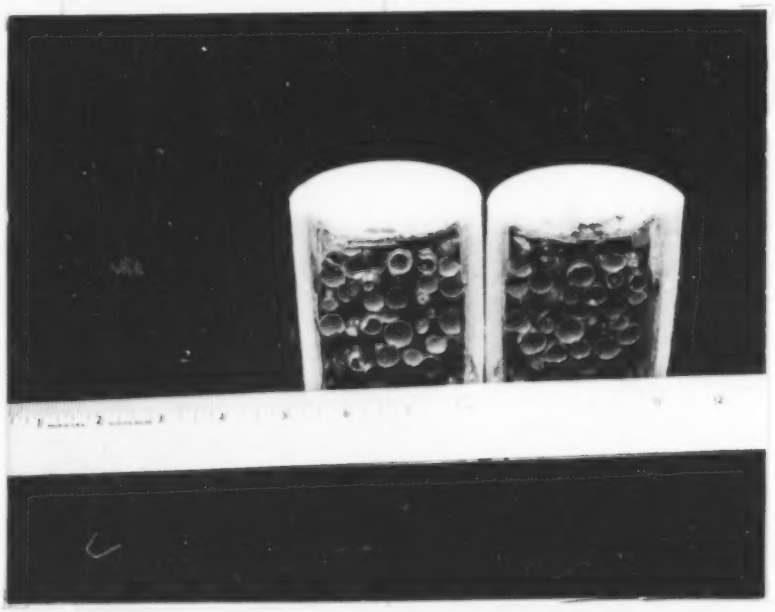

Figure B-9. Cross-section of encapsulate holding thin-walled, hollow plastic balloons.

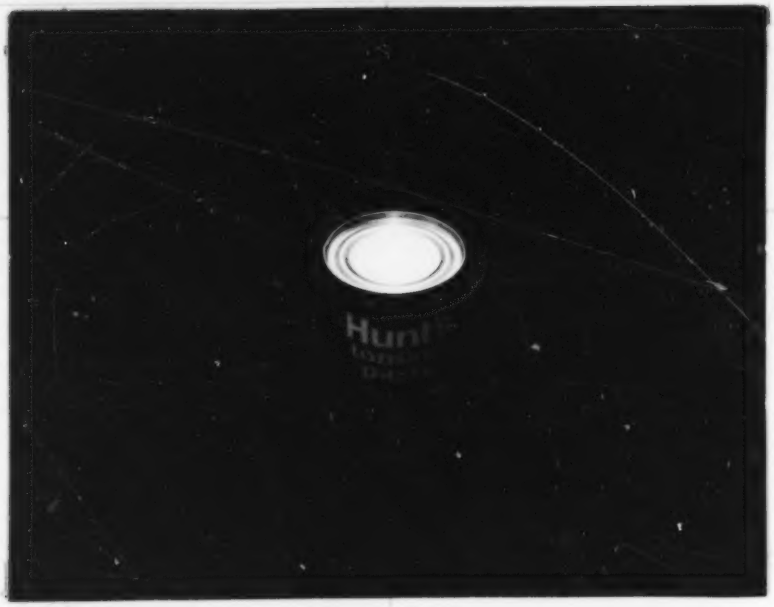

Figure B-10. Container selected for encapsulation simulating 55-gal drum holding liquids.

Figure B-11. Container fitted into mold for fashioning encapsulate.

Figure B-12. Encapsulate of container.

APPENDIX C

MECHANICAL TESTING

A. Tensile

1. All tests were conducted on an Instron Universal Testing Instrument, Model 2230.
2. Testing was conducted in accordance with ASTM-D-638, Type IV.*
3. Testing speed was 50.8 mm (2 in.) per minute.
4. Experimentation was conducted in two steps

   Step I - Compression molding polyethylene alone under normal fabricating condition. Specimens derived from this material were prepared and subsequently tested.

   Step II - Entailed fabricating specimens containing an inner wall of one-ply glass reinforced with resin (approximate width = 0.0035 in.) and a 1/4-in. polyethylene outer jacket.
5. P.E. specimens were fabricated in the fashion described in Section 6.
6. Refer to statistical data on tables: Standard deviations were calculated based on five test specimens as called for by ASTM-D-638.

B. Compression

1. Testing speed = 5.08 mm (0.20 in.) per minute.
2. Testing was performed on the Reihle testing machine, Model FS 160.
3. Tests were based on ASTM-C-39-71, but modified to accommodate specimen size.

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*Type IV specimens were modified to accommodate the actual size of polyethylene ensembles.

APPENDIX D

ATOMIZATION SOURCES FOR ATOMIC ABSORPTION ANALYSIS

The graphite furnace was used for determination of ppm levels of Cd, Cr, Cu, Ni, Pb and Se. The equipment consists of a hollow graphite tube surrounding the atomic absorption beam. The graphite tube is connected to a power source and amperage controller so that the tube may be brought to a temperature of 100^{0} to 3000^{0}C very rapidly (0.5 to 10 sec.). By inducing current in this technique the samples are taken through three temperature stages, 1) the drying phase to remove the solvent matrix, 2) the ashing phase in which organic interferences are volatilized from the dry samples, and 3) the atomization phase in which the element of interest is atomized for absorption in the A.A.S. beam.

As shown in Table 6, a wide variety of dry, ash, atomation temperatures and times may be employed depending on the particular element. In determining the elements Cr, Cu, and Ni, the sample needs simply to be injected "neat" into the graphite tube and the temperature phases set to obtain optimum results.

The elements Cd and Pb require the addition of equal volumes of sample and a solution of 0.2% ammonium molybdate in 1.0% nitric acid to the graphite tube because of the volatility of the two elements[4]. Selenium, which is another very volatile element, is determined by adding equal volumes of sample and a 1000 ppm Ni ($asNiNO_3$) in 1.0% nitric acid.

The mercury content was determined by a cold vapor trap method. The mercury in the sample is reduced to the elemental state using a 10% stannous chloride solution and aerated from the system into the UV cell. The mercury vapor absorbs the UV (254nm) according to its concentration in the fixed cell path. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

Arsenic determination entails the reduction and conversion of arsenic to its hydride in acid solution with $SnCl_2$ and metallic Zn. The volatile hydride is swept from the reaction vessel[2] in a stream of argon and introduced into an argon-hydrogen flame in an atomic absorption spectrophotometer. The hydride is subsequently decomposed and its concentration monitored at its resonant wavelength, 193.7 nm.

Antimony is detected when inorganic antimony reacts sequentially with potassium iodide, stannous chloride, and sodium borohydride to form stibine. The stibine is removed from solution by aeration and swept by a flow of nitrogen into a hydrogen diffusion flame, where it is determined by atomic absorption at 217.6 nm.